PROCESSING, CHARACTERIZATION AND SLIDING WEAR BEHAVIOR OF FUNCTIONALIZED CARBON NANOTUBE REINFORCED EPOXY MATRIX COMPOSITE

A THESIS SUBMITTED

IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE DEGREE OF

Master of Technology

in

Production Engineering

By

Anurag Singh

Department of Mechanical Engineering

National Institute of Technology, Rourkela

Odisha, India - 769008

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C E RT I F I C A T E

This is to certify that the thesis entitled "PROCESSING, CHARACTERIZATION AND SLIDING WEAR BEHAVIOR OF FUNCTIONALIZED CARBON NANOTUBE REINFORCED EPOXY MATRIX COMPOSITE", submitted by MR. ANURAG SINGH bearing Roll no. 212ME2302 in partial fulfillment of the requirements for the award of Master of Technology in the Department of Mechanical Engineering, National Institute of Technology, Rourkela, is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other university/institute for the award of any Degree or Diploma.

Date: 02/06/2014

Dr. S. Biswas
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Rourkela.

Dedicated To My Parents and Sister

Acknowledgement

Though only my name appears on the cover of this dissertation, a great many people have contributed to its production. I owe my gratitude to all those people who have made this dissertation possible and because of whom my master's experience has been one that I will cherish forever

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Last but not the least, I owe more than thanks to my family members which includes my parents and an elder sister for their support and encouragement throughout my life. Without their support, it is impossible for me to finish my education seamlessly.

Abstract

The wear of parts because of friction is becoming the major problem for replacement of any product. The present research work is aimed to minimize the specific wear rate by fabricating the composite material by using multi walled carbon nanotube (MWCNT) as reinforcement. In this study the MWCNT is functionalized and then it is mixed with epoxy matrix composite by employing the chemical functionalization. Various characterization tests are done in order to predict various properties, nature and structural morphologies of MWCNT by Fourier Transformed Infrared Spectroscopy, X-Ray Diffraction, Thermo Graviemetric Analysis, Differential Scanning Calorimetry, and Scanning Electron Microscope. Composite is then prepared from pristine as well as functionalized MWCNT by varying its concentration. Sliding wear tests are done at different concentration and wear surface morphology is inspected by optical microscope. Results of functionalized MWCNT composite were compared with neat epoxy and pristine MWCNT composite and outstanding improvements was noticed for specific wear rate at a concentration of 0.5 wt. %.

Keywords: carbon nanotubes, dispersion, functionalization, sliding wear, tribology.

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Chapter 1 – Introduction

1.1 Background and Motivation

As the growing need of world is increasing day by day, it is therefore becoming impossible for the individual to sustain in the present resources that the world have. So, the need for the development of new material arises. In order to develop a new material there are two things that should be kept in mind one is the innovative product design and second is its requirement in use. These two things lead to the innovative material designed to accomplish the growing need of society. Scientists are focussing their research to develop innovative materials as per the use for specific application. Innovative material research includes developing material either by making internal changes or by making external changes. Internal changes include alloying, heat treatment. External changes include reinforcing the material with fibres, rods, whiskers, particles etc. These both methods result in an improved property of a material. Materials are primarily selected for their mechanical, physical and thermal properties to ensure proper functioning, when several other materials are suitable and no other requirement are to meet than cheapest material is usually selected. In modern world these aspects are very well accounted by the composite materials as they can be very strong and stiff, at the same time very light in weight. In composites it is possible to attain combination of properties not achievable with metals, ceramics or polymers alone. As the properties of composite materials can be tailored made, complex parts can be easily made in a single process thus reducing number of steps required and these materials can be devised for special applications. The material is designed according to its ultimate requirement. In this thesis a composite material is developed keeping these things in mind and its designated area of application.

1.2 Composite Material

First question that arises in our mind whenever we hear some new term is what is it? So, the composite materials, as defined by the Komundari: “Composite material form a material system composed of a mixture or a combination of 2 or more macro-constituents that differ in form and chemical composition and are insoluble in each other” [1]. Other definition as defined by Reinhart: “Composite materials are macroscopic combination of 2 or more distinct materials having discrete and recognizable interface separating them” [2].

Composite generally consist of a matrix material and reinforcement material. Matrix material can be defined as the material that holds the reinforcement in place. When load is applied to the composite material, matrix shares the load with reinforcing element. Whereas, the reinforcing element is a material that provides strength to matrix, it is the major load bearing element. Reinforcement can be majorly classified in four ways as explained below:-

a) **Fibers**: - Fibers can be continuous and discontinuous. Those running throughout the length of composite are called continuous fibers and chopped ones are called discontinuous fibers. Figure 1 shows the various arrangements of fibers in a matrix.
b) **Particulate**: - Particulate can be in the form of spheres, particles having different faces, particles with different shapes. Different kinds of particulate can be seen in Figure 2.
c) **Whiskers**: - Whiskers are single crystals which have widely varying morphology and dimensions. Figure 3 shows different types of whiskers.
d) **Layers**: - Reinforcement are as a continuous layers in the matrix, this can be seen in Figure 4.

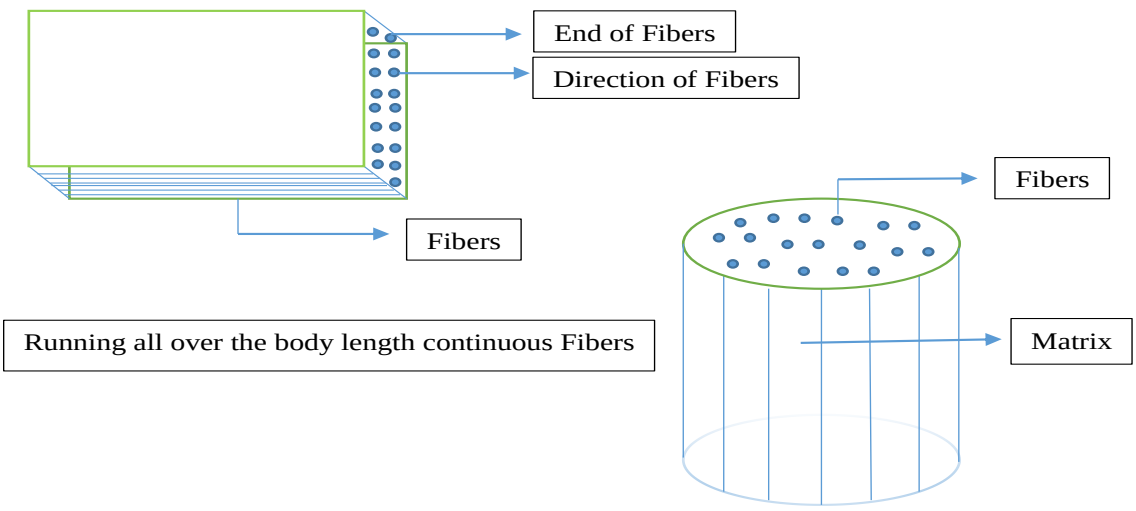

Figure 1 Arrangement of fiber in a matrix

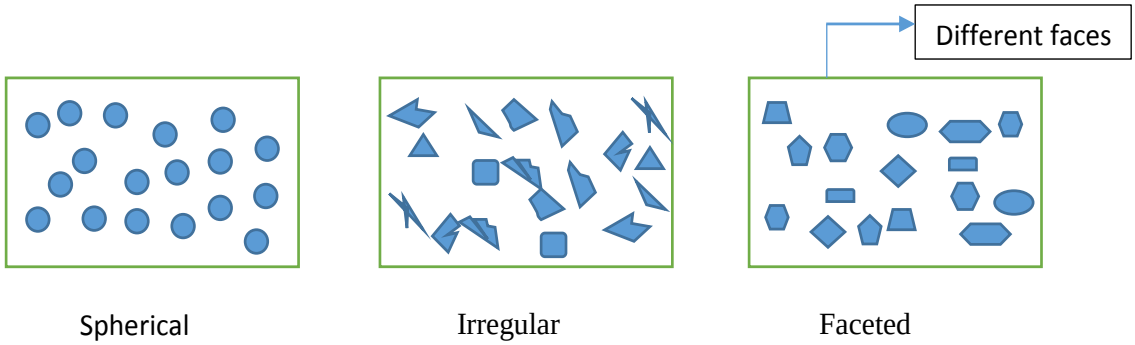

Figure 2 Types of particulate in a matrix.

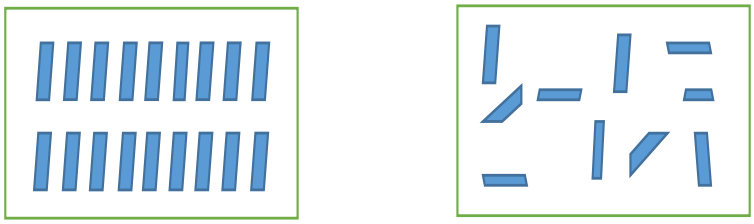

Figure 3 Different types of whiskers in a matrix material

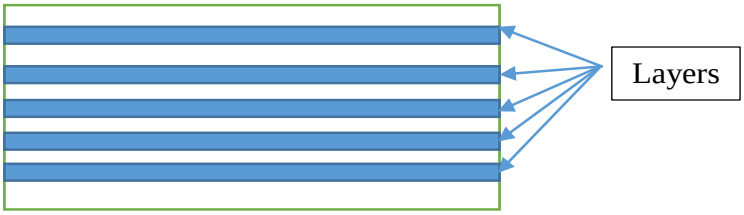

Figure 4 Reinforcement as continuous layer in a matrix

In Figure 5 broad classification of composite as matrix and reinforcement is given. Further, Matrix material can be classified into three categories they are:-

Ceramic Matrix

Metal Matrix

Polymer Matrix

The polymers can be of two types as thermoplastic polymer and thermosetting polymer. Thermoplastic polymer are the material that cures reversibly and softens on heating, when heated above the glass transition temperature or melting point and becomes hard after cooling. It has a linear or branched molecular structure. Properties of thermoplastic polymer include high strength and toughness, chemical resistance, good durability, self-lubrication, transparency and water proofing. Some of the thermoplastic polymer materials are Acrylonitrile Butadiene Styrene (ABS), Polycarbonates (PC), Polyethylene (PE), Polyether ether ketone (PEEK). Polyvinyl Chloride (PVC) etc. Thermoplastic polymer are used to manufacture dashboard, toys, electrical products, bearings, gears, ropes, glass frames, hoses, sheet etc. are just to name few.

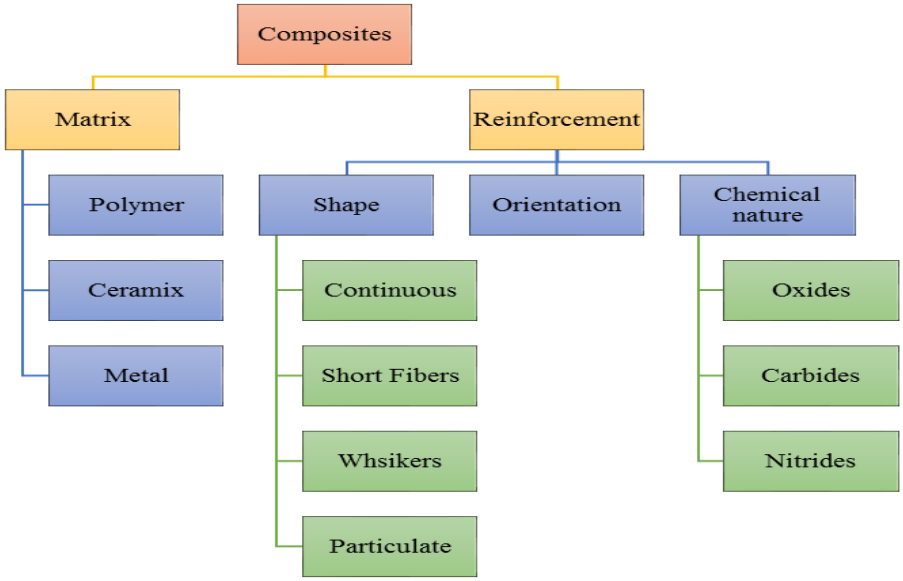

Figure 5 Broad classification of composite as matrix and reinforcement.

Thermosetting polymer is the materials that cures irreversibly and becomes permanently hard and rigid after curing. These cannot be re-melted but continuously heating for a long time leads to degradation and decomposition. Thermosets has a three dimensional network of covalent intermolecular bonds commonly called as cross linked structure. Properties of thermosetting polymers are stiff, tough and durable due to large molecular structure. They have good electrical and thermal insulation, chemically resistant. To name some of the thermosets are Alkyds, Allylies, Bakelite, Epoxies, Phenolic, Polyester, Polyurethane etc. Applications in which these are used are electrical equipment, motor brush holders, printed circuit boards, circuit breakers, encapsulation material, kitchen utensils, handles, knobs, spectacle lenses etc. Figure 6 shows the breakdown of polymer matrix composite explaining different polymers and fillers, which also shows different kinds of thermosetting and thermoplastic polymer material.

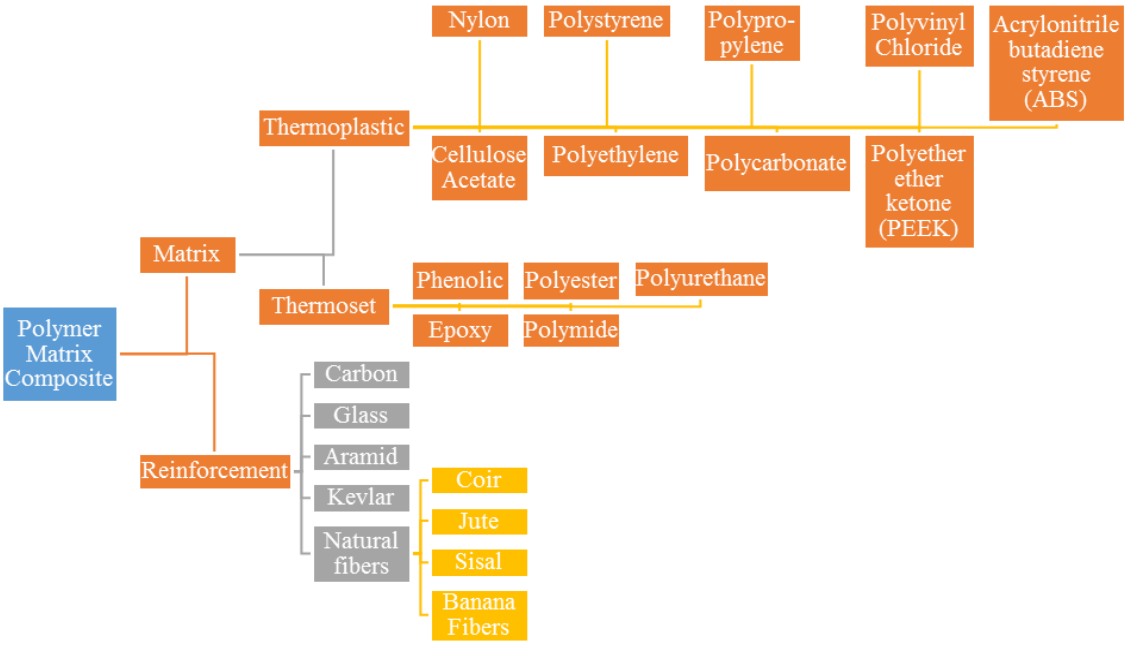

Figure 6 polymer matrix composite.

1.3 Nanotechnology

It all started with a famous talk by a physicist Richard Feynman " There's plenty of room at the bottom" at the California Institute of Technology in an American Physical Society meeting (CalTech) on December 29, 1959, it was only after a decade that the term nanotechnology was coined by Professor Nario Taguchi. Nanotechnology concerns at the size of about 1 to 100 nm. It includes all structures of nanometre range and at microscopic level they also becomes the part of nanotechnology as they are made up of single atom and are considered as a Nano scale subsystems, thus nanotechnology is the study of enormously small things and its application in various fields of science. Nanostructured materials are considered to be consisting of single phase or multiphase polycrystalline solids the size of which varies up to few nanometres. The use of nanotechnology is growing in various areas as in Physics, Chemistry, biology, medicine which leads to discovery of new departments in the field of research like Nano-physics, Nano-medicine, Nano composites, Nano-architecture etc. In this thesis work is conducted in the field of Nano composites. Nano

composites can be defined as those composite materials in which at least one of the phases or filler dimensions like length, width, thickness are in the nanometre range i.e.1 to 100 nm.

1.4 Carbon Nanotubes Fundamentals

Carbon nanotubes are working as an excellent filler in the field of Nano composites, but before going into detail about the carbon nanotubes, first discuss the basic building block from which it is made:-

1.4.1 Carbon

Carbon is the chemical element with symbol C and has an atomic number 6, which means on an atomic orbital it occupies $1s^2$, $2s^2$ and $2p^2$ atomic orbital. It has the ability of hybridizing in sp, sp^2 and sp^3 forms. C_2H_2 is hybridized in sp^2 form. Fullerene, graphene, carbon nanotubes are sp2 hybridized and diamond is sp3 hybridized. From our study point of view sp^2 hybridization is important. Figure 7 shows sp^2 hybridization.

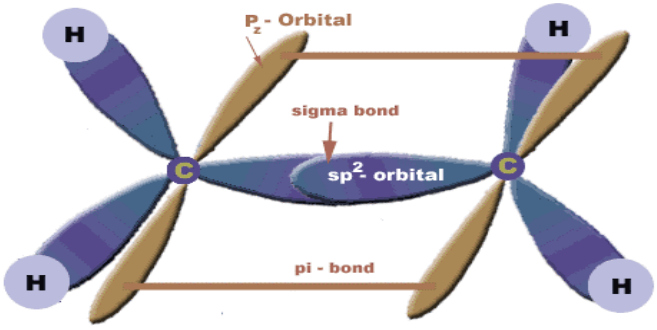

Figure 7 sp2 Hybridization in carbon *[3]*

In this hybridization there is involvement of one s and two p orbitals, here one electron in s orbital gets promoted to the 2p orbital. Three hybrid orbitals are created which are equal in energy but has lower energy than p-orbitals on combining the above said three orbitals. 2p orbitals are orthogonal to the planes. Here, π and π* denotes the bonding and anti-bonding orbital. Fullerene, Graphene, Carbon Nanotubes are all sp^2 hybridized [4].

1.4.1.1 Fullerene

Fullerene generally is in the form of hollow sphere. Therefore, these are also named as "Bucky Balls". They have a resemblance of the soccer ball. The fullerenes were discovered in 1985 by the Smalley research group in the Rice University while conducting experiments on vaporization of carbon by laser pulses.

Buckminsterfullerene C-60 was named after Richard Buckminster Fuller a popular architect, whose geodesic dome is constructed on the same structural principles. Figure 8 shows the Fullerene C-60 molecule which contains 60 vertices and 32 faces (20 hexagons and 12 pentagons).

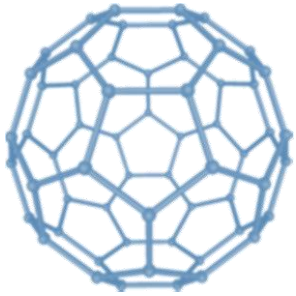

Figure 8 Buckminsterfullerene or C-60 molecule *[5]*.

1.4.1.2 Graphene

Graphene is a one atom thick sheet of graphite. It was only in 2004 that it was discovered. It is having a two dimensional structure and carbon are bonded with sp^2 hybridization. Figure 9 a) shows the graphene sheet, it can be resembled as if one story of a graphite structure (as in Figure 9 b)) is cut to form a single layer of graphene [6].

a) b)

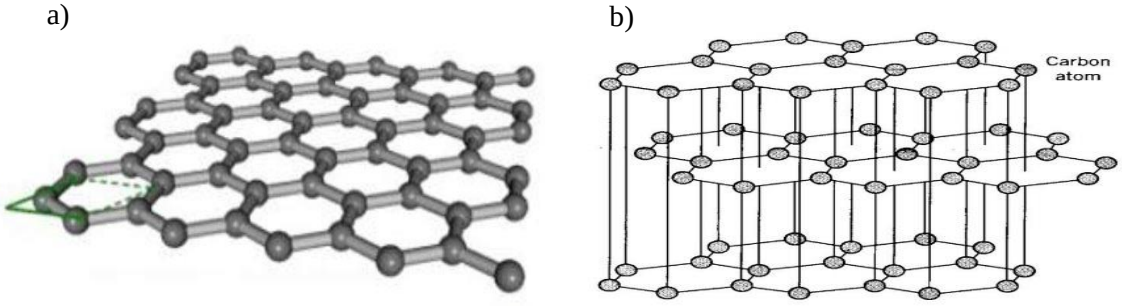

Figure 9 a) Graphene *[7]* b) Graphite *[8]*

1.4.1.3 Carbon Nanotubes

Carbon Nanotubes are a long one dimensional structure made up of carbon in which carbon atoms are sp^2 hybridized. These can be visualized as a graphene sheet which is rolled to form a cylinder as shown in Figure 10. Carbon nanotubes were first synthesized by Sumio Iijima in 1991 [9]. Nanotubes were comprised of several tens diameter of graphite shells, with adjacent shells separation of ~0.34nm and diameter of ~1nm with a very high aspect ratio. It was only after two years that Single wall carbon nanotubes were manufactured by Iijima and Ichihashi [10] and Bethune et al. [11]

Carbon nanotubes possess high flexibility, [12] low mass density [13] and large aspect ratio even up to 1000 has been noticed. These nanotubes are stronger than steel, lighter than aluminum, and more conductive than copper. CNT's are the stiffest known fibers which are by far the highest [14].

Carbon nanotubes basically can be classified in two ways:-

1. Single walled carbon nanotubes
2. Multi walled carbon nanotubes

1.4.1.3.1 Types of carbon nanotubes

1. **Single walled carbon nanotubes**: - These can be imagined as wrapping a one atom thick sheet of graphite which is known to the researchers by the name graphene. Thus, to form a seamless cylinder. Many SWCNT's have a diameter close to 1nm and length of the tubes can be thousand times larger. Journet et.al. have claimed to manufactured SWCNT's of length up to the orders of few centimeters [15].

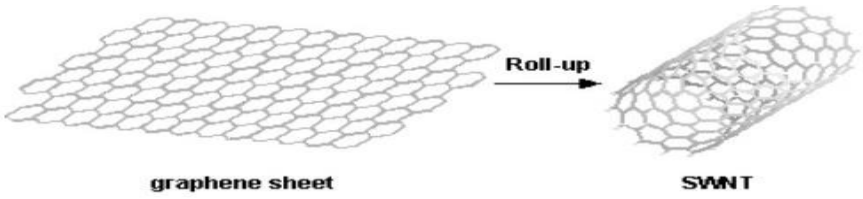

Figure 10 single walled carbon nanotube as visualized from graphene sheet *[16]*

2. **Multi walled carbon nanotubes**: - It consists of multiple layers of graphite which are rolled on to themselves to form a tube. Structure of MWCNT's can be defined by two models which are also shown in Figure 11 and can be visualized.

a. Russian doll model: - sheets of graphene are arranged in concentric cylinders.

b. Parchment model: - a single layer of graphene is rolled around itself, which resembles a rolled up newspaper or scroll of parchment.

Figure 11 a) Russian doll model *[17]* b) parchment model for MWCNT's c) scroll of parchment

3. **Double walled carbon nanotubes**: - It can be visualized as a multi walled carbon nanotubes with just two concentric cylinders as shown below in Figure 12.

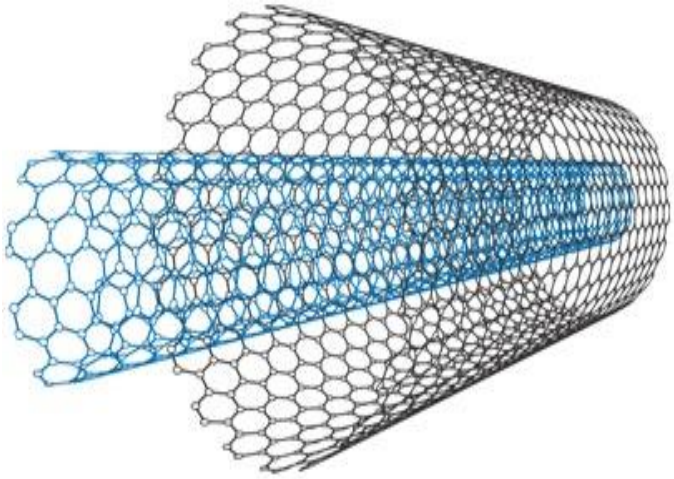

Figure 12 Double walled carbon nanotubes *[18]*

1.4.1.3.2 CNT structural orientation

Carbon in the carbon nanotubes can be arranged in different types. They have different structural patterns of carbon atoms in it. These orientations of carbon hexagonal structure can be named as like armchair, zigzag, chiral. This is represented in Figure 13.

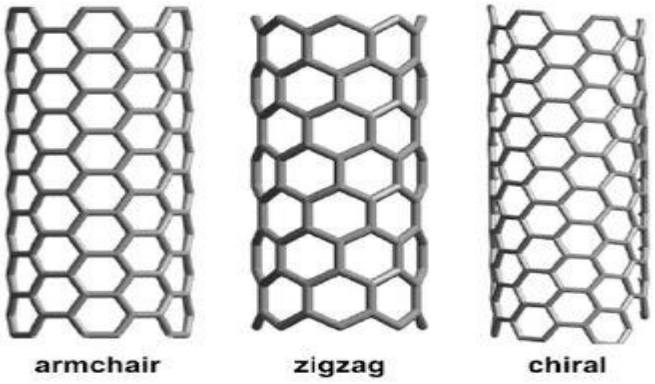

Figure 13 Different orientation of CNT molecule. *[19]*

1.4.1.3.3 Manufacturing of CNT

There are three main methods by which CNT's are manufactured, they are:-

a. Chemical Vapour Deposition (CVD)
b. Electric Arc Discharge Method
c. Laser Ablation Method

1.5 Chemistry involving Functionalization

CNTs functionalization process is very important in order to improve the dispersion and to enhance the bonding between the CNTs and the surrounding matrix, by providing the specific type of the functional group that will bond with the matrix. For Epoxy matrix these functional groups are important as given below. They will increase the dispersion and interfacial bonding of CNT's in epoxy matrix:-

a) Amino ($-NH_2$) Group
b) Carboxyl (-COOH) Group

Other functional groups are not that successful.

During chemical functionalization sulphuric acid creates a defect site on the walls of carbon nanotubes on which nitric acid attacks and oxidizes them. Thus, grafting the carboxylic group on the walls of carbon nanotubes which changes the sp^2 hybridization into sp^3 hybridization. This increases the dispersion behaviour of CNT as hydrophobic nature of CNT changes into hydrophilic which makes them easier to disperse. Composites based on chemically modified nanotubes have shown the best mechanical results because functionalization enables significant improvement in both dispersion and stress- strain transfer. Functionalization is used to increase the dispersion of nanotubes in solvents.

Functionalization is done in order to improve [20]:-

i) Dispersion of CNT in polymer matrix to uniformly distribute load.

ii) Functionalization of CNT's is required to form strong covalent bond between CNT and polymer matrix.

For functionalization following points should be kept in mind [21]:-

a) Higher the curvature of CNT more reactive will be the site.

b) Ends of nanotubes are more reactive than the sidewalls of CNT.

c) Nanotubes synthesized with defects are more susceptible to functionalization than those have no defects.

d) Smaller diameter nanotubes will be easier to functionalize.

e) Carboxylic acids are extremely useful functional group for many reactions; they can be reacted with alcohols to make esters and reaction with primary amines to make amides.

f) Longer nanotubes are more difficult to mix with polymer.

Dispersion of CNT in polymer matrix is affected because of following reasons:-

a) Surface energy of CNT is significantly different from that of matrices as CNT may not have chemical affinity to organic matrices. Thus, dispersion of CNT's into matrices is biggest obstacle.

b) Seamless surface of CNT cannot provide physical interaction between CNT and matrix.

There are two major drawbacks of chemical functionalization [22]:-

a) During functionalization reaction along with the damaging ultra sonication process, a large number of defects are created on CNT sidewalls and in some cases CNT's are fragmented into smaller pieces. Carbon hybridization changes from sp^2 to sp^3 which results in severe degradation in mechanical properties.

b) Concentrated acids or strong oxidants are often used for CNT functionalization which are environmental unfriendly.

There are four variables that control the acid functionalization process these are:-

i) **Concentration of acids used**:- (Ratio between sulfuric acid and nitric acid for grafting carboxyl group)

Sulfuric acid creates small defect site on the surface of CNT's and nitric acid will oxidize these defects sites by providing a functional group, a carboxylic group.

Effect of Concentration of acids used:-

a) Increase in the concentration of sulfuric acid will introduce more defects (roughness) on CNT surface. Therefore, by changing the concentration, level of functionalization can be controlled.

b) Increasing the concentration of sulfuric acid will introduce more defects on CNT surface; this excessive surface treatment could break, damage, dissolve or deteriorate mechanical properties of CNT.

ii) **Temperature of solution**: - Reaction temperature should be maintained constant over functionalization process.

Effect of Temperature of solution:-

a) High temperature would increase the reaction speed.

b) Low temperature could slow it down.

iii) **Duration of acid treatment of the CNTs**.

Effect of Duration of acid treatment of the CNTs:-

a) The more the CNTs stay in contact with the acids, the more the functionalization will be introduced to the CNTs surfaces.

iv) **CNTs/acids ratio**:- (It will affect the severity of the functionalization process)

Effect of CNTs/acids ratio:-

a) If the CNTs concentration is relatively high, then more acids will be needed to complete the chemical reaction.

1.5 Tribology

Tribology is the study of friction, wear, and lubrication of interacting surfaces which are in relative motion [23]. The study of tribology is important as most of the energy goes as friction. Wear is the common encountered problem in industry as it has led to the replacement of parts that are in continuous interaction. Medalia reported that 63% of wear has contributed to the total cost of industries [24]. But wear cannot be eliminated but can be substantially minimised. Burwell classified the five important types of wear as (1) Abrasive wear (2) Adhesive wear (3) Erosive wear (4) Surface fatigue wear (5) Corrosive wear [25]. Tribological properties of any material depends upon the material being tested,

test condition viz. temperature, pressure etc. grit size of the erodent, equipment and atmosphere.

1.7 Organization of Thesis

Remaining chapters in this are organized as follows:-

Chapter 2: Includes a literature review which provides a summary of knowledge already available by involving the issues of interest. It presents the research works on tribological properties by various researchers, which includes carbon nanotube as reinforcement and is divided according to availability of literature.

Chapter 3: Includes a description of the raw materials used in the research and various test procedures. It presents the details of fabrication and characterization of the composites under investigation and also an explanation of the Taguchi experimental design.

Chapter 4: Presents the results that are obtained during various characterization tests and these are discussed in detail. Result of wear test done on pristine MWCNT composite and functionalized MWCNT composite has been presented by employing Taguchi orthogonal array and wear morphology is also described by optical microscopy.

Chapter 5: Provides summary of the findings of this research work, outlines specific conclusions drawn from both the experimental and analytical efforts and suggests ideas and directions for future research

Chapter 2

Literature Survey

- On Mineral Oils
- Coating
- Metal Matrix Composite
- Polymer Matrix Composite
- Knowledge Gap
- Research Objective

Chapter 2 – Literature Survey

Sole aim of this literature review is provide background information on the past work that is done in the field of tribology while taking carbon nanotube as reinforcement. The review has been done in the chronological order keeping each section in mind. It has been divided in the following sections:-

1. Mineral oils
2. Coating
3. Metal matrix composite
4. Polymer matrix composite

At the end of this chapter a knowledge gap is presented along with the objectives of the present research work is also defined.

2.1 On Mineral Oils

Pottuz et al. [26] used carbon nanotubes as an additive to lubricating base oil and noticed that by the addition of only 1 wt% of CNT, there is tremendous fall in friction coefficient and wear. For this observation contact pressure of 0.83 GPa is needed to obtain this result, which indicates necessity for a structural modification of material. High fraction of catalyst particles and low amount of iron after friction marks the formation of a protective layer which may be similar to Ni-doped DLC-like material, which is well known for its tribological properties. Diana et al. [27] showed that when compared with base oil and base oil having a classic commercial antiwear (AW) and extreme-pressure (EP) additive to a mineral base oil. Single walled carbon nanotubes showed lower friction coefficient. Thus, It was concluded that single walled carbon nanotube reduces friction at an optimum concentration of 0.5 %. Scars and grooves were filled on the contact surfaces and the friction is reduced as revealed after analyzing SEM, AFM, and XPS data. Cursaru et al. [28] investigated the tribological properties of single walled carbon nanotubes,

functionalized single walled carbon nanotubes, multi walled carbon nanotubes and fullerenes and found that when 0.2% of single walled carbon nanotube is added, it leads to 13% reduction in friction coefficient and wear rate is reduced by 63% on disk and 47% wear rate is reduced on ball. Optimum concentration is found out to be at 0.2% and on increasing concentration, increase in friction coefficient and wear rate on ball and disk is noticed, agglomeration of nanotubes is given as reason. Functionalized single walled carbon nanotubes, fullerenes, and multi walled carbon nanotubes are shown to have as suitable antiwear additives for vegetable oil. They also found that 0.2% functionalized single walled carbon nanotubes would be suitable for formulation of bio lubricants. Subrajit [29] presented in his research that after adding the nanoparticles to the lubricant, the properties such as viscosity, viscosity index, fire point and flash point is enhanced along with the load carrying capacity and antiwear properties which are considered to be important from lubricant point of view. Coefficient of friction is found to be minimum at a concentration of 0.5%. On increasing the concentration there is reduction in lubricating properties. But, antiwear and load bearing capacity has been increased. This is explained as due to the formation of agglomerate and precipitation of nanoparticles, it has resulted in loss to frictional surface

2.2 Coating

Wang et al. [30] observed that Ni based electro less coating showed higher wear resistance and lower friction coefficient than Ni-P composite coating under unlubricated condition. Deposition of Ni based CNT composite coating by electro less plating on a medium carbon steel after treatment at 673K for 2h. With the increase in concentration of CNT the friction coefficient of composite coating deceased it is because of the self-lubrication and unique topological structure of CNT. While, wear rate increased with the increase in concentration of CNT. This trend follows up to 0 to 11.2 % concentration after

that wear rate decreases because CNT gets conglomerate in the matrix. At higher concentration cracking and spalling occur on the worn surface during wear process. Chen et al. [31] coated the cu matrix composite with Ni–P electro less coating and it showed higher wear resistance and lower friction coefficient when compared to Ni–P–SiC and Ni–P–graphite coating. Copper displayed inferior properties on comparison with CNT copper matrix composite. The reason for this change as explained by him as because of the excellent mechanical properties and extraordinary topological structure of hollow nanotubes, CNT acts as spacers as during experiment it may be released from matrix preventing asperities from contacting the surface.

2.3 Metal matrix composite

2.3.1 Copper as a Matrix Material

Dong et al. [32] concluded that oxidation wear is the key wear mechanism for cu matrix carbon nanotube composite when working under dry sliding condition. The carbon film can decrease the friction and wear rate. Therefore, carbon nanotube copper composite exhibit better wear properties as compared to carbon fiber copper composite. Optimum nanotube content is between 12% and 15% in which coefficient of friction and wear rate is minimum and with the increase of concentration wear rate and friction decreases. Tu et al. [33] reported that copper matrix CNT composite exhibited lower coefficient of friction on comparison with copper alloy. Coefficient of friction decreases with the increase of concentration. At higher concentration of CNT, severe surface damage occur because of its high porosity, thus decreasing the wear resistance. Kyung et al. [34] used molecular level process for homogeneously dispersing carbon nanotubes with copper matrix to form Nano composites. With the increase in the concentration of carbon nanotubes hardness and wear resistance increased for CNT reinforced cu matrix composite.

2.3.2 Aluminum as a Matrix Material

An et al. [35] fabricated the alumina CNT composite by hot pressing the alumina CNT composite powder which is manufactured in catalytic decomposition of acetylene over alumina powder impregnated with iron catalysts, CNT concentration ranged from 0 to 12.5%. For 0 to 4% CNT concentration wear and mechanical properties are enhanced, but beyond 10% there is decrease in mechanical properties because of poor cohesion between Carbon nanotube and Aluminum matrix. Decrease in friction coefficient is explained as due to the lubricating properties of carbon nanotubes and due to the rolling motion of CNT's in boundary between specimen surface and its counterparts. Lim et al. [36] reported that by the introduction of tape casing followed by lamination and hot pressing extraordinary tribological properties were achieved. Up to 12% concentration wear loss and friction coefficient decreased effectively. He explained it as because of effective dispersion and by the effect of carbon nanotube densification and wear debris. Yamamoto et al. [37] prepared the alumina/MWCNT composite by SPS technique. Steady state friction coefficient value reached to as low up to 0.33. But, no improvement to strength and toughness is made, it may be due to agglomeration of MWCNT and also due to the weak interface between multi walled carbon nanotube and alumina matrix. Ahmad et al. [4] reported improved wear resistance and low coefficient of friction for composite reinforced at 2, 5, 10 wt% concentration which were under low and moderate sliding loads. While, for high sliding loads, at 10 wt% concentration it showed lower wear resistance.

2.4 Polymer Matrix Composite.

2.4.1 Polystyrene (PS)

Yang et al. [38] investigated that CNT's has reportedly increased the wear resistance and decreased the coefficient of friction, wear tests were done under dry sliding condition. PS-CNT composite were fabricated by "in-situ" polymerization process. At 1.5wt%

concentration lowest wear rate is noticed. High stiffness and toughness were reported to have an increased wear resistance and self-lubricating property is also reported to have an impact.

2.4.2 Polyphenylene (PP)

Cho [39] reported that by adding 0.2 or 2 vol% MWCNT's, the specific wear rate of polyphenylene sulphide composite reduced considerably. Flexural strength and hardness is reported to increase up to the addition of 0.2 to 1 vol% multi walled carbon nanotubes, but on further addition it gets decreased because of the presence of voids, structural defects and CNT agglomeration. It was also examined that uniform dispersion plays a key role in tribological and flexural strength of any composite.

2.4.3 Polyethylene (PE)

Zoo et al. [40] reported that up to 5 % addition of CNT wear of Ultra-high-molecular-weight polyethylene (UHMWPE) is considerably reduced but there is increase in frictional coefficient is reported which is due to the increase of shear strength of softer UHMWPE. It was concluded that by addition of CNT, it can acts as a promising additive for polymer based composites that require good wear resistance. Wei et al. [41] reported that pure UHMWPE composite film showed poor wear resistance and high friction coefficient when compared with MWCNT/UHMWPE composite films, it is because of the addition of multi walled carbon nanotubes, a new microstructure is formed which has strong interfacial bonding between MWCNT's/UHMWPE composite films.

2.4.4 Poly Tetra Flouro Ethylene (PTFE)

Chen et al. [42] fabricated CNT/PTFE composite with concentrations varying from 2.5 to 30 vol%. Self-lubrication of CNT's were explained when friction coefficient decreased on increasing the concentration. At 20 vol% CNT/PTFE composite exhibit lowest wear rate. During contact CNT's acts like spacers because of which there is increase

in wear resistance and decrease in friction coefficient. Vail et al. [43] observed that at all loading there is an increase in wear resistance by more than 2000%. There is 50% increase in coefficient of friction than that of unfilled PTFE at low speed.

2.4.5 Polyimide (PI)

Cai et al. [44] founded that for carbon nanotube reinforced polyimide composite there is lower friction coefficient and wear loss when it was compared with neat polyimide under dry sliding. Wang et al. [45] observed that the tribological properties of PI composite in which combined inclusion of nano-Si3N4, short carbon fibers, and graphite showed most effective results. Filled PI composite exhibited better friction and wear behavior when working under oil lubrication and worse when working under water lubrication as compared to dry lubrication.

2.4.6 Epoxy

Dong et al. [46] used a friction and wear tester to study the tribological properties of multi walled reinforced epoxy matrix composite. It was observed that MWCNT's increased the wear resistance significantly while decreasing the friction coefficient. These significant tribological properties attributed to high mechanical strength and topological tubular structure of MWCNT's. To achieve these results a uniform dispersion is the foremost requirement and self-lubrication of MWCNT's leads to better tribological properties. Sulong et al. [47] concluded that addition of carbon nanotube to pure polymer showed less weight loss and it significantly increases the tribological properties by performing a dry wear test, self-lubrication of CNT was stated to be the major factor. Functionalized CNT will have the better tribological properties than the pristine CNT as stated. Zhang et al. [48] observed that surface area of carbon nanotubes plays a major role in wearability of composite. When the coverage area ratio of CNTs to matrix on a pin surface Rc/m increased beyond 25% than wear rate can be reduced by a factor of 5.5, this

increase is because CNT is exposed to the sliding surface which prevented the epoxy to come under the direct contact area thus protecting it. Material removal is via brittle fracture when the value of Rc/m is low while a ductile fracture is observed under high Rc/m. CNT can be fragmented significantly during the contact sliding in wear test. Chen et al [49] explained that dual asymmetric centrifuge can be used to effectively increase the dispersion, it produces sufficient shear stress to disintegrate the agglomerates while preventing the damage of CNT. CNT's treated with coupling agent or nitric acid gives very good wear results when dispersed by the speed mixer. Wang et al [50] explained that aligned CNT-epoxy showed improved wear resistance, lower friction coefficient, high hardness and higher young's Modulas than the neat epoxy. Zhang et al. [51] used the 3, 6-diamino-1, 2, 4- and 5-tetrazine compounds to undergo reaction with the carbon nanotubes and successfully attached the amide group onto the walls of carbon nanotubes. This grafted amino group reacts with the epoxy group to undergo a chemical bond which improves the interfacial properties. This leads to the decrease in the friction coefficient to about 6.5%, wear rate is reduced to about 71.4%. Thus, tetrazine influenced the tribological properties very effectively. Cui et al. [52] finds that including MWCNT's with epoxy resin is an efficient way to improve the wear resistance of epoxy resin, it also shows a lower friction coefficient and wear rate when compared with the neat epoxy, wear rate and friction coefficient increased significantly with the increase of applied load and sliding speed, composite having composition of 0.5wt% amino MWCNT's shows the lowest wear rate and it decreases by 41.3%. Santos et al. [53] noticed that superior mechanical properties were observed at an effective nanotube reinforcement of 0.75%. Glass transition temperature increases by 10ºC when MWCNT added to the matrix. For tribological properties 0.25% addition of MWCNT's showed 24% decrease in wear rate and 17% reduction if coefficient of friction.

2.5 Knowledge Gap

From the literature review done above, it reveals the following gap in the literature reported above:-

1. Although, a great deal of work has been done on the pristine MWCNT based composites, however very little work has been reported on the wear behaviour of functionalized MWCNT composite. Thus, the need for chemical functionalization by carboxylic group arises.
2. CNT's have taken part in every research field extensively. But, the area of the tribological properties, specifically sliding wear test of carbon nanotubes remains unexplored by the researchers.

2.6 Research Objective

1. To find the parameters for functionalization of multi walled carbon nanotubes by carboxylic group.
2. To confirm the grafting of carboxylic group on to the walls of MWCNT's by FTIR spectrometer.
3. To check whether the functionalization condition were smooth enough so as not to distort the conjugated structure of MWCNT by XRD.
4. To find the optimum parameters of sliding wear test by Taguchi design.
5. To study the effect of concentration on wear surface morphology and specific wear rate.

Chapter 3

Materials and Methods

- Introduction
- Materials
- Chemical Functionalization
- Composite Fabrication
- Physical Test
- Scanning Electron Microscope
- Fourier Transformed Infrared Spectroscopy
- X-Ray Diffraction
- Thermo-Gravimetric Analysis
- Differential Scanning Calorimetry
- Wear Test
- Optical Microscope

Chapter 3 - Materials and Methods

3.1 Introduction

This chapter gives an insight of various materials used during the research work and methods that are employed for the successful completion of research. This provides detailed description about various characterization techniques that are done on the pristine carbon nanotubes and after functionalization on the functionalized carbon nanotubes and also various parameters taken. Nano composites fabrication detail and after fabrication the insight of sliding wear test by using the Taguchi experimental design and statistical explanation by analysis of variance (ANOVA) on the "Pin on Disk" test rig.

3.2 Materials

3.2.1 Matrix Material

For the preparation of composite materials, matrix material can be of basically three types as disused in earlier chapters as metal matrix, ceramic matrix and polymer matrix. In this study polymer matrix is chosen as they are easily available, can be fabricated with ease, and its properties can be tailored according to the need of applications very effectively when compared with the ceramic and metal matrix. Further polymer matrix can be classified as thermoplastic polymer and thermosetting polymer.

Commonly employed thermosetting polymers are epoxy, polyester, vinyl ester and phenolic. Of all these materials epoxy has been used widely for many advanced composites, with carbon nanotubes a large amount of work has been published which shows excellent mechanical and thermal properties also at high temperature, wide range of viscosity is available with the epoxy, for carbon nanotube a low viscosity matrix material is needed in order to obtain good dispersion. Due to these advantages with the carbon nanotube epoxy

material Lapox L12 is selected. In the following sections a detailed insight of L12 and hardener K6 will be given.

Lapox L12 is a liquid, unmodified epoxy resin of medium viscosity which can be used with various hardeners. The choice of hardener depends upon the processing methods to be used and on the properties required of the cured composite. Thus, hardener K6 is chosen as it has a low viscosity as it is of prime importance with carbon nanotubes. Being reactive it gives a short pot life and rapid cure at ambient temperatures.

Specification of resins and hardener

a) Lapox L-12

i) Epoxide equivalent gm/eq. 182-192

ii) Epoxy value eq. / kg 5.25-5.5

iii) Viscosity at 25ºC 9000-12000 mPa.S

b) Hardener K-6

i) Visual appearance pale yellow liquid

ii) Refractive index at 25 ºC 1.4940-1.5000

iii) Water content 1% max

Processing parameters

Lapox L-12 100 parts by weight

Hardener K-6 10-12 parts by weight

Viscosity at 20ºC 5000-8000 mPa.S

Pot life at 20 ºC 0.5-1 hrs.

Lapox L-12 and hardener K-6 can be easily mixed at room temperature.

Curing range at various temperatures

25 ºC	14-24 hrs.
80 ºC	1-2 hrs.
100 ºC	15-30 minutes
140 ºC	5-10 minutes
180 ºC	instantaneous

Lapox L-12 is known by the conventional name and its IUPAC name is 4, 4'-Isopropylidenediphenol, oligomeric reaction products with 1-chloro-2, 3-epoxypropane it belongs to the epoxide family [54]. The epoxy resin and corresponding hardener are procured from Ciba Geigy India Ltd.

3.2.2 Filler Material

Multi walled carbon nanotubes have been selected as a filler material. As, it has a very high aspect ratio, Young's Modulas is over 1TPa, estimated tensile strength is around 200 GPa, It is as stiff as Diamond. Since carbon nanotubes have a low density for a solid of 1.3-1.4 g/cm^3, its specific strength is the best of known materials. Thus, these properties are best suited for fabricating reinforced composites. Multi walled carbon nanotubes have been procured from J.K. Impex, Mumbai. Its specifications as mentioned by supplier are given below:-

Purity > 97%

Outer Diameter – 11-20 nm

Length – 10-30 microns

3.3 Chemical Functionalization of Multi-walled Carbon nanotubes

For the chemical functionalization of multi walled carbon nanotubes by carboxyl group, mixture acid is used which is a combination of sulphuric acid and nitric acid. Sulphuric acid with 98 wt. % concentrations and nitric acid with 69-72 wt. % concentration

are taken. Various tests run are done in order to arrive at the appropriate parameters for the chemical functionalization. In the chemical functionalization of MWCNT 100 ml of mixture acid is taken and the following parameters are taken as shown in Table shaded runs are those whose FTIR spectroscopy has been done, as for others conditions were harsh, so it has destroyed the conjugated structure of CNT.

Table 1 Different parameters for chemical functionalization of multi walled carbon nanotubes.

Run	Mixture acid concentration	Time of Heating	Temp. of Mixture	Quantity of MWCNT
1	3:1	1 h	90 º C	100 mg
2	3:1	1 h	90 º C	200 mg
3	3:1	3 h	90 º C	200 mg
4	3:1	1 h	90 º C	300mg
5	3:1	3 h	90 º C	300mg
6	3:1	5 h	90 º C	300mg
7	2:1	1 h	90 º C	300mg
8	3:1	30 minutes	120 º C	300mg
9	2:1	30 minutes	120 º C	300mg
10	1:1	30 minutes	120 º C	300mg
11	3:1	30 minutes	120 º C	1 g
12	2:1	30 minutes	120 º C	1 g
13	1:1	30 minutes	120 º C	1 g
14	3:1	30 minutes	120 º C	800mg

Production of functionalized multi walled carbon nanotubes is done in bulk by the test run 11. Description of how this run is selected is done in results and discussion. Sulphuric acid and nitric acid (Mixture acid) are taken in a 3:1 ratio, 3 parts of sulphuric acid is mixed with 1 part of nitric acid in a beaker and then multi walled carbon nanotube is added to it. Now, the mixture is sonicated in an ultrasonicator for 15 minutes for proper mixing of CNT with the mixture acid. After that the sonicated mixture is poured in a double neck round bottom flask whose one neck has been equipped with thermometer for measuring temperature accurately and other neck is occupied by the condenser arrangement so as to heat the solution under reflux, thus not allowing CNT to escape. The solution is

heated in a heating mantle under reflux for 30 minutes at a temperature of 120º C. After heating the mixture it is allowed to cool to a room temperature which is followed by washing and filtering of MWCNT by the PTFE membrane filter. Material and pore size of the filter is chosen carefully as mixture acid will dissolve the material of the filter. So, Poly Tetra Flouro Ethylene (PTFE) is selected as the filter material as it will sustain the acid, pore size is selected at 0.22 micron for the CNT to be filtered from the solution and the size of the filter is selected as 25mm diameter according to our set up for filter. PTFE membrane filter is procured from S.S. filters, Mumbai. Filter is kept in a filter assembly which is connected to a vacuum pump so as to create vacuum for easily facilitating the filtering of CNT. Filtered CNT is washed several times by the deionized water (5 washing cycles are enough for neutralising the pH level of MWCNT) and pH is measured by litmus paper, it must become neutral for further processing. Now, the filtrate obtained is kept in a pettry dish and put in an oven at 60º C for 24 h for the CNT to dry. FTIR is done so to confirm the grafting of carboxyl group onto the walls of MWCNT's and XRD is done to check the structure of MWCNT's after chemical treatment. Experimental setup for chemical functionalization in fig 14 shows the heating mantle which is used for heating the mixture placed in double neck round bottom flask, whose one neck is equipped with thermometer for measuring the temperature precisely and other one is equipped with condenser.

3.4 Composite Fabrication

Composite is fabricated by pristine MWCNT and functionalized MWCNT. By keeping the concentration of MWCNT in matrix in mind, MWCNT is taken and mixed with acetone which is used as a solvent. MWCNT's were taken in a small beaker and sonicated for 15 minutes, then the epoxy is added into the mixture and it is mechanically stirred for 2 minutes which is followed by sonication for 4h, the dispersed mixture thus obtained is magnetically stirred for 1h at 70ºC so as to completely dispersed the MWCNT into the

epoxy matrix, this will completely vaporize the acetone present in the mixture which is added so as to obtain better dispersion by decreasing the viscosity of the mixture, now the hardener is added into this mixture and again it is mechanically stirred for 2 minutes then it is sonicated for 10 minutes after which it is poured into the syringes so as to get the specimen of 10mm diameter and 30mm height, height can be adjusted in the syringe by adjusting the plunger.

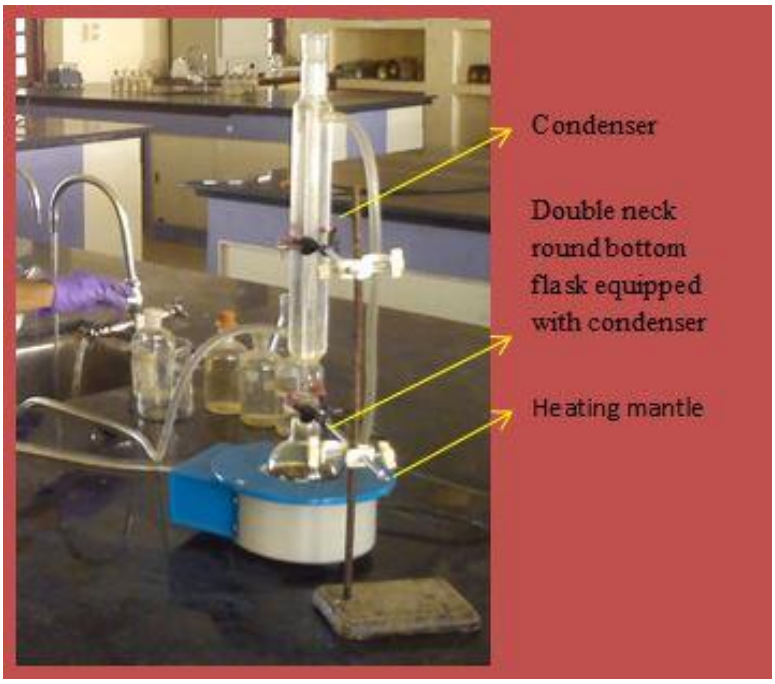

Figure 14 experimental setup of chemical functionalization

Table 2 Designation of Composites

Composites	**Composition**
0	Pure Epoxy
1P	Pristine Multi walled carbon nanotube (1wt %) + Epoxy (99 wt. %)
1.5P	Pristine Multi walled carbon nanotube (1.5wt %) + Epoxy (98.5 wt. %)
2P	Pristine Multi walled carbon nanotube (2 wt. %) + Epoxy (98 wt. %)
3P	Pristine Multi walled carbon nanotube (3 wt. %) + Epoxy (97 wt. %)
1F	Functionalized Multi walled carbon nanotube (1wt %) + Epoxy (99 wt. %)
1.5F	Functionalized Multi walled carbon nanotube (1.5wt %) + Epoxy (98.5 wt. %)
2F	Functionalized Multi walled carbon nanotube (2 wt. %) + Epoxy (98 wt. %)
3F	Functionalized Multi walled carbon nanotube (3wt %) + Epoxy (97 wt. %)

Then, samples are allowed to cure at room temperature for 2 days. This procedure is followed for both pristine and functionalized MWCNT. The detailed composition and label of the composites are presented in Table 2. Samples after curing is taken out of the syringes and polished.

3.5 Physical Test

In physical test, density is calculated which is a very important property in terms of application in which this composite is going to incorporate; density is of prime importance for weight sensitive applications. Density of composite material is affected by the weight fraction of matrix and filler material. There is always the difference in the values of theoretical and actual density of composite. Due to the presence of voids actual density will always be less than the theoretical density, thus void content should be as minimum as possible. But, voids will be always present in the material, we cannot diminish them but we can always minimise them. The theoretical and actual density of the composite is measured and the difference is explained as the void content. Theoretical density of the composite is measured by weight fraction of matrix material and filler material and is calculated by the formula given by Agarwal and Broutman [55] as given below

$$\rho_{th} = \frac{1}{\frac{W_f}{\rho_f}+\frac{W_m}{\rho_m}} \tag{3.1}$$

Where, $\rho_{th} = Theoretical\ density$

$W_f = Weight\ fraction\ of\ fibre$

$\rho_f = Density\ of\ fibre$

$W_m = Weight\ fraction\ of\ matrix$

$\rho_m = Density\ of\ matrix$

Actual density of various composite at different concentration and neat epoxy is calculated by water immersion technique.

3.6 Scanning Electron Microscope (SEM)

A scanning electron microscope produces images by scanning the sample by the focused beam of electrons; an electron interacts with the samples and produces various signals that contain various information about the surface topography. Generally electron beam is scanned in a raster scan mode. For this research work SEM-JEOL-JSM-6480 equipment has been used as shown in the Figure 15. For SEM sample preparation the surface of the specimen is coated with platinum for 5 minutes prior to the observation at 50kV after that the surface topography of the multi walled carbon nanotubes is studied [56].

Figure 15 SEM setup.

3.7 Fourier Transform Infrared Spectroscopy (FTIR)

Vibrational spectrum of the molecule is considered to be a unique physical property and is a characteristic of the molecule. Infrared spectrum can be used as tool for identification of spectrum of unknown materials, as it acts a finger print and compares the data with the previously recorded reference spectra. This is the basic principle of computer generated spectral search. The term frequency is used for band/peak position and it is expressed in units of wavenumber (cm^{-1}). The average modern infrared instrument records

spectra from an upper limit of around 4000 cm^{-1} (by convention) down to 400 cm^{-1} as defined by the optics of the instrument (commonly based on potassium bromide, KBr). For this reason, when a spectral region is quoted in the text, the higher value will be quoted first, consistent with the normal left-to-right (high to low cm^{-1}) representation of spectra.

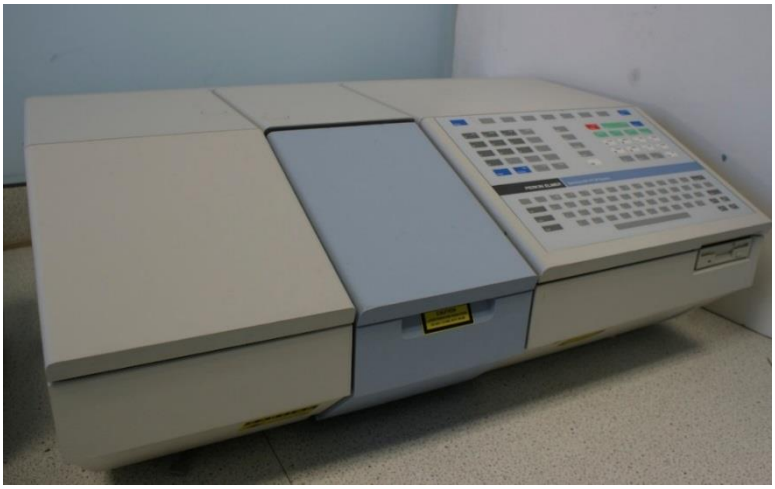

Figure 16 FTIR instrument (Perkin Elmer, USA)

FTIR instrument used here is Perkin Elmer, USA (as shown in Figure 16) which is installed in the chemistry department of NIT Rourkela, pallets of carbon nanotube are made with kBr with the help of a palletizer which is shown in fig. 17. Because of the black character of carbon nanotubes, a weak concentration of nanotube is used in a kBr powder. As it exhibits strong absorbance and it is than not possible to distinguish from the background noise. Sample for the measurement of FTIR were grinded and a very low concentration is used approximately 0.02%. Fig. shows the FTIR spectrum of functionalized MWCNT.

Figure 17 Palletizer

3.8 X-Ray Diffraction (XRD)

XRD is a tool used to identify the atomic and molecular structure of the crystal, it provides identification of phase of crystalline materials and it also provides information on the unit cell's dimension. XRD is a non-destructive analysis technique which gives the unique fingerprint of Bragg's reflection associated with the structure of crystal, crystal structure is reflected as layers of atoms arranged in a plane. When x-rays are scattered from a crystal lattice, peaks of scattered intensity are observed which correspond to the following conditions:

The angle of incidence = angle of scattering.

The path length difference is equal to an integer number of wavelengths.

The condition for maximum intensity contained in Bragg's law above allow us to calculate details about the crystal structure, or if the crystal structure is known, to determine the wavelength of the x-rays incident upon the crystal. This can be visualized from Figure 18. It is explained by Bragg's law as: -

$$n\lambda = 2d\ sin\theta. \qquad (3.2)$$

Where, λ is wavelength of x ray.

Θ is the angle between the incident rays and surface of the crystal.

d is spacing between layers of atoms.

Constructive interference occurs when n is an integer. When n is an integer 1, 2, 3 etc.

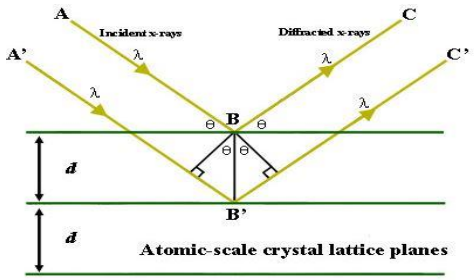

Figure 18 Incident and reflected rays in XRD. *[57]*.

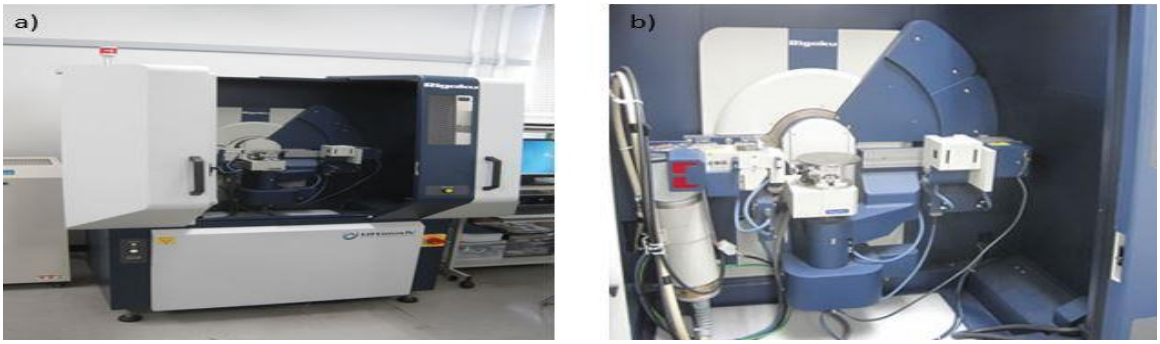

Figure 19 XRD instrument Rigaku Japan/Ultima-IV. a) Setup of XRD b) Place for holding the slide during the XRD.

X-Ray Diffraction (XRD) is carried out using Rigaku Japan/Ultima-IV equipment as shown above in Figure 19. Pristine MWCNT is taken in a slide and the following parameters are chosen

Source of wavelength - Cu Kα X ray

Wavelength - 1.5418Å

Step size of 0.02º

Range - 5º -90º (2Θ)

10º per step per minute

Same parameters were chosen for the functionalised MWCNT and the XRD is done.

3.9 Thermo Gravimetric Analysis (TGA)

TGA is a technique by which the mass of sample is observed with reference to the temperature as it is heated or cooled. It consists of a simple pan which is attached with a precision balance, this pan resides inside a furnace and weight loss is monitored as a function of time. Environment of the sample is filled by sample purge gas. Gas may be inert or reactive and it flows over the sample, exits as exhaust.

In this research work TGA (Shimadzu, *Japan*) equipment is used. Sample is heated from the room temperature to the 700ºC in a nitrogen atmosphere with a gas flow rate of 20ml/min with a heating rate of 10ºC/min.

Grafting ratio can be calculated from the graft ratio formation by the given formula [58].

$$R = \frac{\frac{x}{M_a}}{\frac{(1-x)}{M_c}} \times 100 \qquad (3.3)$$

Where, $x = weight\ loss\ of\ the\ sample.$

$M_a = Molecular\ weight\ of\ carboxylic\ group$

$M_c = Atomic\ weight\ of\ carbon.$

From this the graft ratio of MWCNT after functionalization is calculated.

3.10 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) is a very common technique of measuring glass transition temperature. Glass transition temperature is a temperature at which amorphous solid begins changes into the amorphous liquid. If the fibre is semi-crystalline, its crystalline melting temperature Tm also can be measured. DSC includes heating or cooling a specimen at a constant rate in ºC/min. This instrument measures heat capacity which is calculated by precisely measuring amount of heat added or removed from the sample. Heat capacity is directly proportional to the amount of amorphous materials that changes from amorphous solid to amorphous liquid. In the region of glass transition

temperature there is steep increase in the heat capacity and where glass transition temperature is absent heat capacity increases linearly with temperature [21]. Differential Scanning Calorimetry is performed with DSC 822 (Mettler Toledo) equipment as shown below in Figure 20. The heating process was scanned from 30-150°C with a heating rate of 10°C/min under nitrogen atmosphere. It is performed on neat epoxy, pristine MWCNT's composite, and functionalized MWCNT's composite, latter two are prepared at different composition. The prepreg were kept in an aluminium pan and weight of the samples is taken in between 10 to 30 mg.

Figure 20 DSC setup and computer attached to it for monitoring the data.

3.11 Wear Test

Sliding wear test is carried out on the pin on disk machine (DUCOM; TL-20), experimental setup of which is shown in Figure 21, it is used to evaluate the sliding wear behaviour of the composite. Here, the abrasive paper of grit size 220 is used. Abrasive paper is glued to the disc with the help of Araldite which is an epoxy based standard adhesive, then it is kept overnight. The disc is mounted on to the disc holder, disc is

connected to the DC motor and has the range of RPM from 0 - 600 rev/min. Sliding occurs between the stationary pin and the rotating disk, load is applied on a pin by the help of pulley arrangement, and applied load has a range from 5N to 20N. Samples is mounted on the pin holder and after each run were cleaned using acetone and then dried for 2 minutes and weighted to an accuracy of ±0.01mg before and after the sliding wear test using electronic balance as shown in Figure 22, so as to calculate the specific wear rate after the experiment.

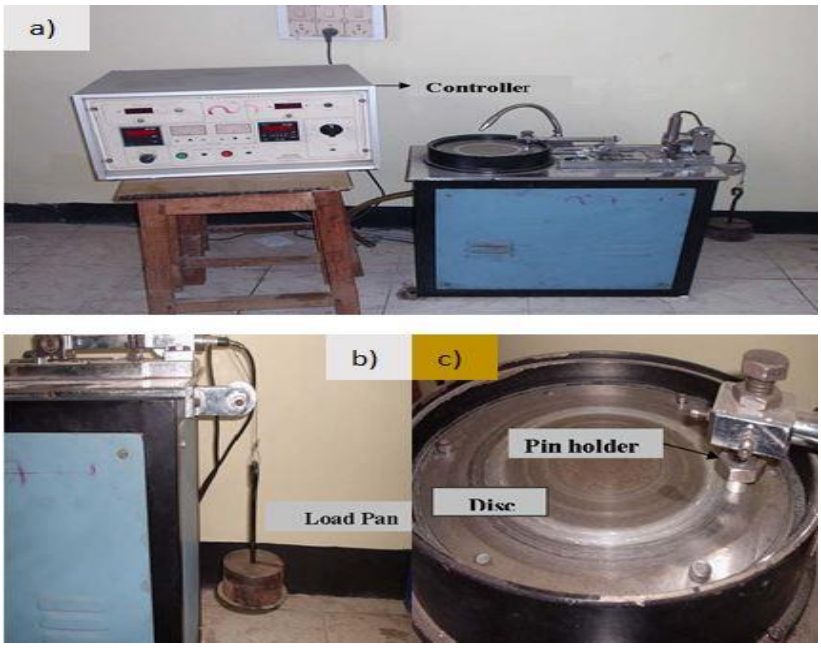

Figure 21 a) Experimental setup of pin on disk arrangement b) Loading arrangement c) Pin holder and disk.

3.12 Taguchi Design

For any research as test procedures are generally expensive and also time consuming. Therefore, the need to minimise the number of tests is of foremost importance. In this context, Taguchi Method provides systematic and efficient approach to do the experiment and to arrive at optimum design parameters for performance and cost. This method designs the specially constructed Table known as orthogonal array. Use of orthogonal array significantly decreases the number of experiments. The conclusion which

is drawn from these orthogonal arrays is valid for the entire control factors at all the levels together. Selection of control factors for setting the orthogonal array is most important.

Figure 22 Precise weighing scale.

Literature review reveals that parameters viz., concentration, RPM, Load, and Sliding Distance influence the specific wear rate of polymer composite extensively. Therefore, impacts of these four parameters are studied using L16 orthogonal design. Various factors selected and their levels are shown in Table 3. The tests are conducted as per the experimental design. The experimental observations are transformed into signal-to-noise (S/N) ratios. There are several S/N ratios available depending on the type of characteristics such as:

'Smaller-the-better' characteristic:

$$\frac{S}{N} = -10\, log \frac{1}{n} (\sum y^2) \tag{3.4}$$

'Nominal-the-better' characteristics:

$$\frac{S}{N} = -10 \log(\sum \frac{\bar{y}}{S^2}) \tag{3.5}$$

'Larger-the-better' characteristics:

$$\frac{S}{N} = -10\, log \frac{1}{n} (\sum \frac{1}{y^2}) \tag{3.6}$$

Where n is the number of observations, y is the observed data, Y is the mean and S is the variance. The S/N ratio for minimum erosion rate comes under 'smaller is better' characteristic, which can be calculated as logarithmic transformation of the loss function by using Eq. (3.4).

Table 3 various factors in Taguchi design.

	Levels				
Control Factor	I	II	III	IV	Units
A. RPM	100	200	300	400	Rev/min
B. Concentration	0	1	2	3	Wt.%
C. Load	5	10	15	20	N
D. Sliding Distance	251.2	376.8	502.4	753.6	m

Taguchi design of experiment is to be applied and design is made according to the 4 factors each of which has 4 levels. The 4 factors that are selected for the design are RPM, concentration, load, and sliding distance. Based on these, L16 orthogonal array is prepared and run are done, which are based on these parameters. Total of 32 numbers of experiments are to be done, 16 for pristine MWCNT composite and 16 for functionalised MWCNT composite respectively. This is shown in Table 4.

3.13 Wear Surface Morphology by Optical Microscopy

After the sliding wear test, optical microscope is used to see the wear surface morphology at different concentration viz. Neat epoxy, Pristine MWCNT composite, Functionalized MWCNT and their specific wear rate is also compared. Samples are taken at a composition of 0.5wt%, 1wt%, 1.5wt%, 2wt%, 3wt%, Optical microscope used is OLYMPUS BX51M optical Microscope. Tests are done at different concentration while keeping load, RPM, sliding distance constant at 10N, 200 rev/min, and 314m respectively.

For keeping the sliding distance constant each experiment is run for 10min., after this the wear surface morphology is studied and compared.

Table 4 L_{16} orthogonal array for pristine MWCNT's composite and functionalized MWCNT's composite for sliding wear test.

S. No.	RPM	Concentration	Load	Sliding Distance
1	100	0	5	251.2
2	100	1	10	376.8
3	100	2	15	502.4
4	100	3	20	753.6
5	200	0	10	502.4
6	200	1	5	753.6
7	200	2	20	251.2
8	200	3	15	376.8
9	300	0	15	753.6
10	300	1	20	502.4
11	300	2	5	376.8
12	300	3	10	251.2
13	400	0	20	376.8
14	400	1	15	251.2
15	400	2	10	753.6
16	400	3	5	502.4

Chapter 4

Results and Discussion

- Tests results for FTIR
- Characterization
- Wear Test

Chapter 4- Results and Discussion

This chapter presents the results that are obtained during various characterization tests and these are discussed in detail. Result of wear test done on pristine MWCNT composite and functionalized MWCNT composite has been presented by employing Taguchi orthogonal array. Wear surface morphology is analysed by optical microscopy after sliding wear test on pin on disk apparatus by employing a Taguchi design.

4.1 Tests results for FTIR

As it is already discussed in previous chapter that total of 14 number of experiments are done in order to arrive at the proper parameters for chemical functionalization. For confirming the grafting of carboxylic group FTIR spectrum is checked. For run number 1, 2, 3, 7, 13 the experimental conditions were harsh which left no residue after functionalization for doing the FTIR and all the CNT were completely lost. Table 5 shows the result of various FTIR spectrums. For the confirmation of grafting of carboxyl group onto the walls of MWCNT there must be the presence of four bonds and these are O-H Bond, C=O Bond, C-O Bond, C-H Bond, the spectrum for these four bonds have as O-H Stretch from 3300-2500 cm^{-1} and from 1440-1395 cm^{-1}, for C=O Stretch it is 1800-1600 cm^{-1}, C-O Stretch varies from 1400-600 cm^{-1}, C-H Stretch varies similar to O-H stretch 3300-2500 cm^{-1} [59] [56] [52] [60], these portions are highlighted in the Table below. If our spectrum lies in this range then it can be said that carboxylic group has been successfully grafted.

Figure 23 shows the FTIR spectrum for run number 4, 5, 6, 8. For run 4 O-H, C-H, C=O bond were not present. For run 5 and 6 C-O bond is absent, and for run 8 C=O bond is not available. Thus, for these runs it is said that grafting of carboxyl group is not successful.

Table 5 Tests result for FTIR after chemical functionalization

Run	O-H Stretch	C-H Stretch	C=O Stretch	C-O Stretch
Required Spectra	3300 to 2800 cm^{-1}, 1440 to 1395 cm^{-1}	3300 to 2500 cm^{-1}	1760 to 1680 cm^{-1}	1400 to 600 cm^{-1}
1	NIL	NIL	NIL	NIL
2	NIL	NIL	NIL	NIL
3	NIL	NIL	NIL	NIL
4	NIL	NIL	NIL	1337.54 cm^{-1}
5	3008.23 cm^{-1}	3008.23 cm^{-1}	1695.83 cm^{-1}	NIL
6	3007.82 cm^{-1}	3007.82 cm^{-1}	1729.93 cm^{-1}	NIL
7	NIL	NIL	NIL	NIL
8	2878.10 cm^{-1}, 2778.12 cm^{-1}, 3004.30 cm^{-1}, 2609.46 cm^{-1}, 3030.78 cm^{-1}, 950.02 cm^{-1}	2878.10 cm^{-1}, 2778.12 cm^{-1}, 3004.30 cm^{-1}, 2609.46 cm^{-1}, 3030.78 cm^{-1},	NIL	1389.63 cm^{-1}, 1321.02 cm^{-1}, 1259.78 cm^{-1}, 1214.03 cm^{-1}, 1098.59 cm^{-1}, 1049.70 cm^{-1}, 950.02 cm^{-1}, 615.33 cm^{-1}
9	3015.93 cm^{-1}	3015.9 cm^{-1}	NIL	951.48 cm^{-1}
10	3002.71 cm^{-1}, 2876.35 cm^{-1}	3002.71 cm^{-1}, 2876.35 cm^{-1}	1745.69cm^{-1}, 1713.65 cm^{-1}, 1647.82 cm^{-1}	NIL
11	3011.37 cm^{-1}, 2776.28 cm^{-1}	3011.37 cm^{-1}, 2776.28 cm^{-1}	1729.49 cm^{-1}, 1712.31 cm^{-1}, 1694.58 cm^{-1}	1230.62 cm^{-1}
12	3009.28 cm^{-1}, 1415.02 cm^{-1}	3009.28 cm^{-1}, 2876.35 cm^{-1}	1694.43 cm^{-1}	NIL
13	NIL	NIL	NIL	NIL
14	3260.61 cm^{-1}, 3198.38 cm^{-1}, 3150.03 cm^{-1}	3260.61 cm^{-1}, 3198.38 cm^{-1}, 3150.03 cm^{-1}	NIL	NIL

Figure 24 shows the FTIR spectrum for run number 9, 10, 12, 14. For run 9 C=O bond is absent. For run 10 C-O bond is unavailable, for run 12 C-O bond is not available. For run 14, two bonds C=O and C-O were not present. Thus, for these runs it is said that grafting of carboxyl group is not successful.

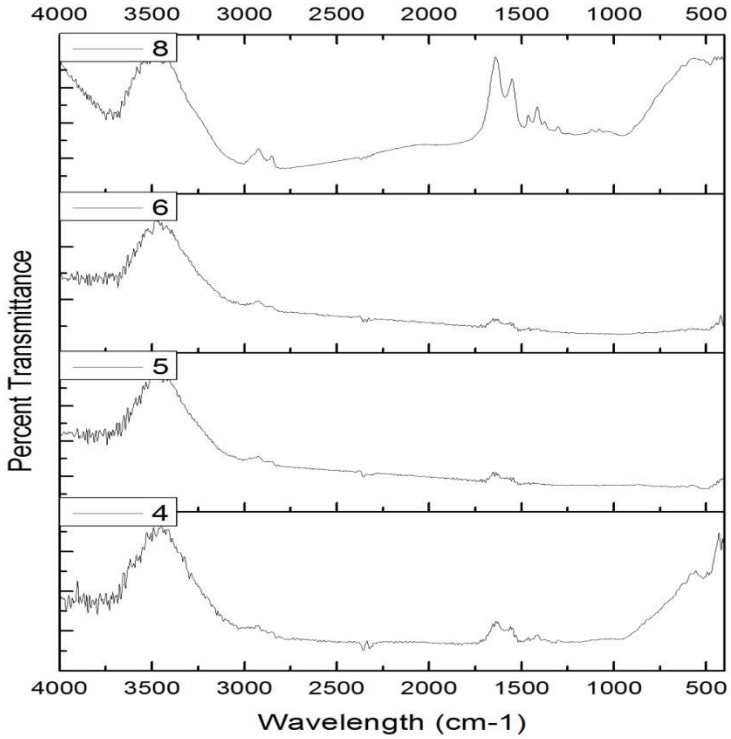

Figure 23 FTIR spectrum for run number 4, 5, 6, and 8.

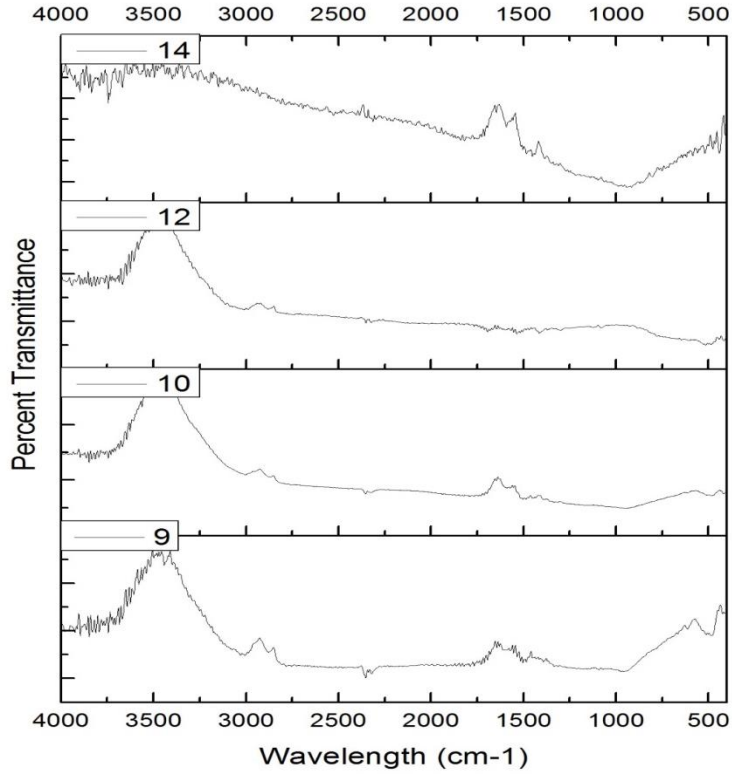

Figure 24 FTIR spectrum for run number 9, 10 ,12, 14.

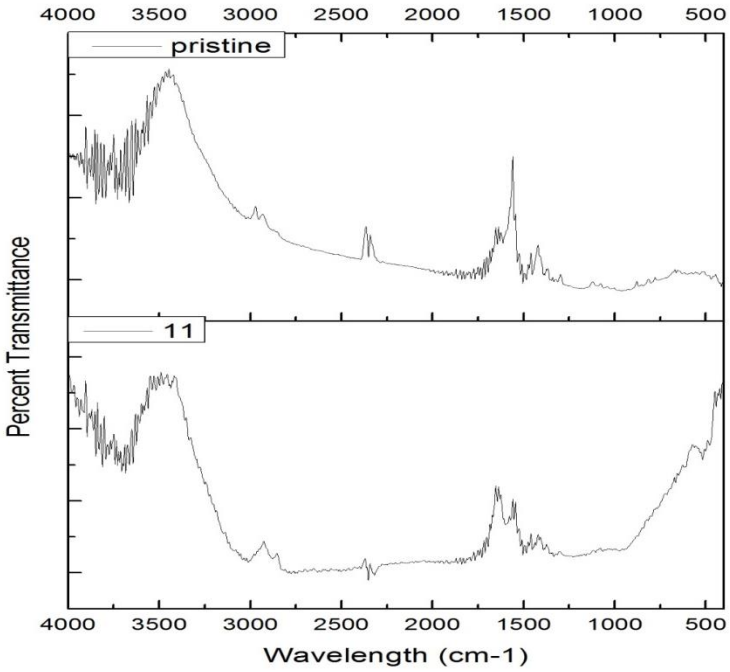

Figure 25 FTIR spectrum for run number 11 and pristine MWCNT.

Figure 25 shows the FTIR spectrum for pristine MWCNT and run 11. Pristine MWCNT were not expected to show any result for grafting and it come out to be likewise. The graph for run 11 shows the O-H peak intensity at 3011.37 cm^{-1} and 2776.28 cm^{-1}, 1433.49 cm^{-1}, C=O peak intensity at 1729.49 cm^{-1}, 1712.31 cm^{-1}, 1694.58 cm^{-1}, C-O peak intensity at 1230.62 cm^{-1}, C-H peak intensity at 3011.37 cm^{-1}, 2776.28 cm^{-1}, which is well within the range for confirming the presence of carboxyl group. Thus, from run 11, the confirmation for the presence of grafting by carboxylic group has been done. Therefore, the parameters for functionalization that are obtained are as – the mixture acid concentration is kept at 3:1, temperature of the mixture is set at 120ºC, time of heating is 30 minutes and 1 g of pristine MWCNT is taken. By taking these parameters successful grafting is obtained and bulk preparation of functionalized MWCNT is done.

4.2 Characterization

4.2.1 Surface Morphology using SEM

In Figure 26 SEM image of the pristine MWCNT is shown, from Figure it can be concluded that as such CNT's are Non-Conducting. As in Figure below for unpolished MWCNT, images are not clear. Thus, platinum coating is done over it. In the polished samples it can be seen that they are arranged in the form of bundles because of the large Vander walls force of attraction between them. MWCNT's are tangled with each other and their diameter is very small. White portion in the image denotes the charge particles.

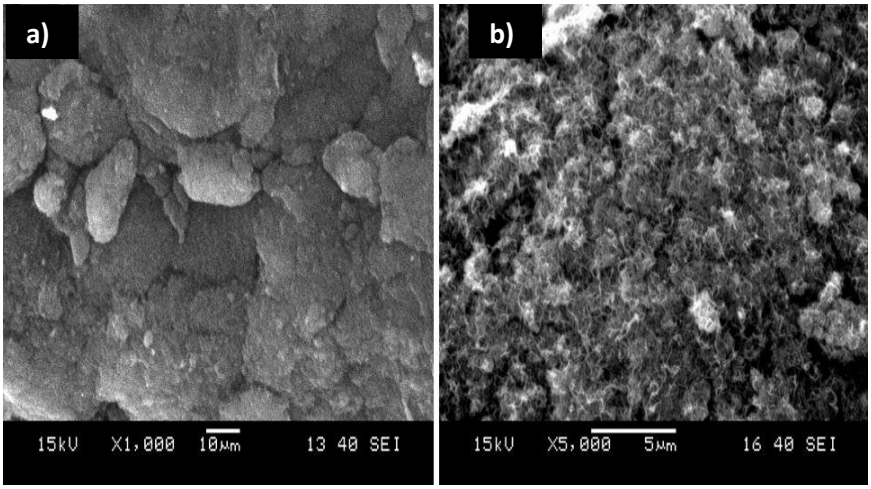

Figure 26 SEM images of a) Unpolished MWNCT's b) Polished MWCNT's

4.2.2 X-Ray Diffraction (XRD)

XRD pattern for pristine MWCNT and functionalized MWCNT composite are shown in Figure 27 and Figure 28 respectively, the diffraction peaks for pristine MWCNT is at 25.728º, 43.26º, 53.70º, 78.15º and for functionalized MWCNT's is at 25.763º, 42.79º, 52.93º, 78.64º. These diffraction patterns can be attributed to hexagonal graphite structure (002), (100), (004), and (110) [61]. This shows that after the chemical functionalization of MWCNT's the structure of MWCNT's is retained after the grafting by the carboxyl group on the surface of MWCNT.

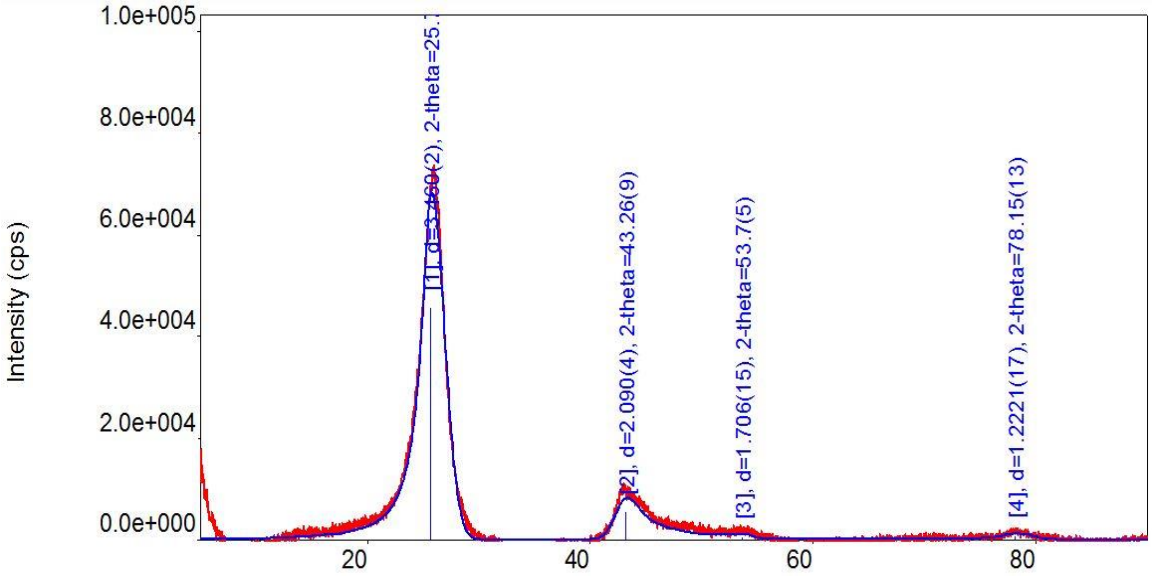

Figure 27 XRD spectrum of pristine MWCNT.

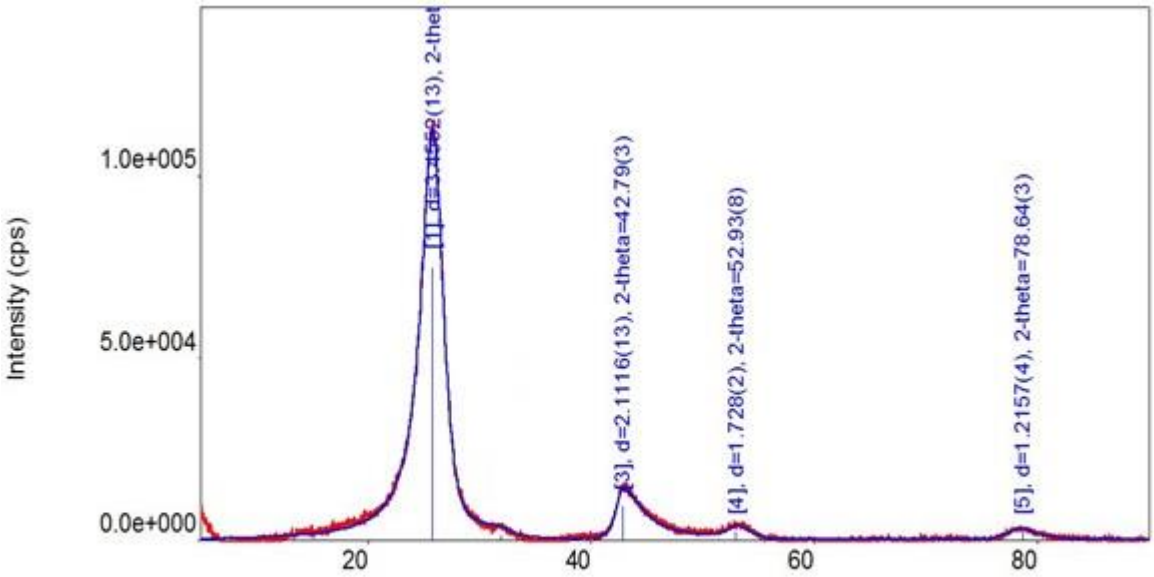

Figure 28 XRD spectrum of functionalized MWCNT.

4.2.3 Thermo Graviemetric Analysis (TGA)

Figure 29 shows the TGA data for two different samples of functionalized MWCNT on which the successful grafting of carboxylic group has been done by chemical functionalization. The graft ratio for both the samples is calculated by the formula given in earlier section and shown in Table 6.

Table 6 Graft ratio of two different functionalized MWCNT samples.

	Sample 1	Sample 2
Weight loss %	29.32%	35.04%
Graft Ratio	11.06%	14.38%

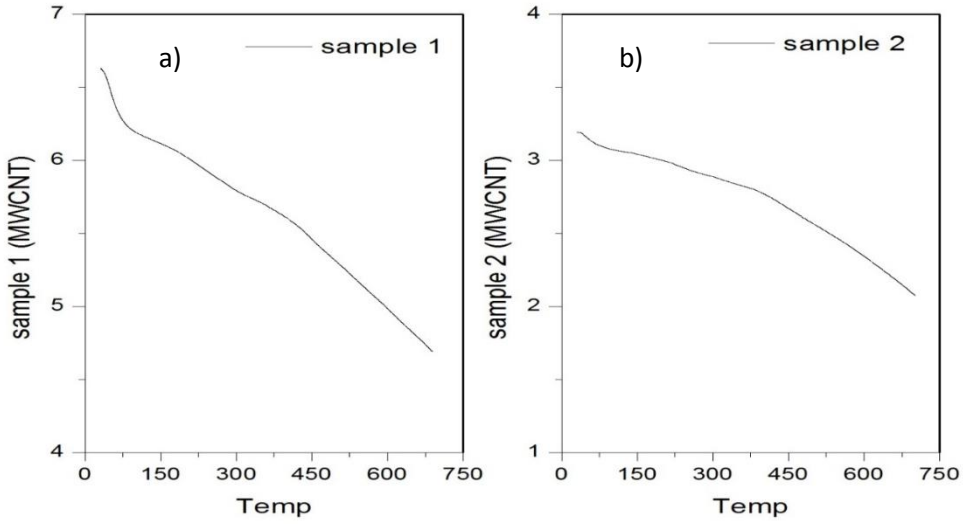

Figure 29 TGA graphs of two different samples of functionalized MWCNT.

Taking average of both the samples for grafting ratio, the grafting ratio comes out to be 12.72%. Thus, it can be said that 12.72 molecule of carboxylic group is present for every 100 carbon atoms.

4.2.5 Differential Scanning Calorimetry (DSC)

Table 7 shows the glass transition onset temperature for pristine MWCNT composite and functionalized MWCNT composite which is analysed at different composition. DSC results have shown no particular trend both for pristine MWCNT composite and functionalized MWCNT composite. Thus, no particular conclusion is drawn from these results.

Table 7 Glass transition onset temperature.

Composition	T_g (onset Temperature in ℃)	
	Pristine MWCNT Composite	Functionalized MWCNT Composite
0	65.15	65.15
0.5	63.18	62.41
1	71.91	59.46
1.5	68.29	67.18
2	66.26	64.66
3	74.36	67.37

4.2.6 Physical Test

4.2.6.1 Effect of concentration on density

The difference in theoretical and experimental densities is mainly due to the presence of voids in the composite. Hence, it becomes essential to certain extent to determine the percentage of voids in the samples prepared. Table 8 shows the theoretical density, experimental density and the corresponding void content. It can be seen that the void fraction in the composites increases with the fibre loading as with the increase of loading effective dispersion of MWCNT is reduced as they tends to agglomerates and it becomes necessary to overcome the Vander walls force of attraction.

4.2.6.2 Effect of functionalization on density

As it is seen that density is decreasing on increasing the concentration. But, it can be noticed that after functionalization the trend for void content gets reduced when compared to the pristine MWCNT's, it is because that after functionalization with the carboxyl group tendency to form agglomerates of CNT's gets greatly reduced and the dispersion of CNT's in epoxy matrix gets enhanced subsequently.

Table 8 Void fraction of MWCNT composite at different concentration.

S. No.	Composites	Theoretical Density (gm./cc)	Experimental Density (gm./cc)	Volume Fraction of voids (%)
1	0	1.15	1.146803	0.278
2	1P	1.153495	1.12228647	2.7056
3	1.5P	1.155251	1.12076343	2.9853
4	2P	1.157012	1.11682916	3.473
5	3P	1.16055	1.11478879	3.9431
6	1F	1.153495	1.12585538	2.3962
7	1.5F	1.155251	1.12651427	2.4875
8	2F	1.157012	1.12265703	2.9693
9	3F	1.16055	1.12228131	3.2975

4.3 Wear Test

Taguchi orthogonal array was designed for finding the optimum parameters for the sliding wear, there are four factors that are influencing the specific wear rate and each factor have 4 levels. L_{16} array for pristine and functionalised MWCNT composite has been prepared as mentioned above in chapter 3, using this the specific wear rate is calculated from the given formula:-

$$Specific\ wear\ rate\ (w_s) = \frac{\Delta V}{\rho \times F_n \times D} \quad (4.1)$$

Where,

$\Delta V = Volume\ Differenece$

$\rho = Density$

$F_n = Normal\ Applied\ Load$

$D = Sliding\ Distance$

Table 9 and Table10 represents how the experiments are carried out by keeping various factors in mind for the pristine MWCNT composite and functionalized MWCNT composite, then the corresponding SN ratios are calculated after the specific wear rate and main effect plot for each factor is drawn by the help of "Microsoft excel". Here, SN ratio for each factor is calculated by taking the mean of SN ratios for that particular factor.

"Smaller is better" approach is followed as we have to minimise the specific wear rate. Figure 30 shows effect of various control factors on the specific wear rate for pristine MWCNT composite, from various graphs between mean of SN ratios and control factors, it can be concluded that specific wear rate comes out to be minimum for 1P composite at 200 rpm, 20N load, and at a sliding distance of 753.6 m. It can be explained as because of long length of pristine MWCNT, tendency to form agglomerates, high Vander wall forces between them. They are not easily dispersed in a polymer solution and on increasing the concentration they tend to become less soluble with the polymer. Thus, on increasing the concentration, there is more specific wear rate.

Table 9 L_{16} Orthogonal Array for pristine MWCNT composite, specific wear rate.

S. No.	RPM	Concentration	Load	Sliding Distance	Specific wear rate	SNRA1
1	100	0	5	251.2	1.21E-03	58.30880822
2	100	1	10	376.8	4.24E-05	87.44641995
3	100	2	15	502.4	4.26E-05	87.4217098
4	100	3	20	753.6	2.48E-05	92.10046649
5	200	0	10	502.4	1.11E-04	79.08716954
6	200	1	5	753.6	2.83E-05	90.96824513
7	200	2	20	251.2	3.01E-05	90.41695621
8	200	3	15	376.8	4.73E-05	86.50365256
9	300	0	15	753.6	4.71E-05	86.54782268
10	300	1	20	502.4	1.33E-05	97.54941952
11	300	2	5	376.8	1.09E-04	79.27200327
12	300	3	10	251.2	8.87E-05	81.04362712
13	400	0	20	376.8	9.72E-05	80.24700848
14	400	1	15	251.2	3.77E-05	88.4694704
15	400	2	10	753.6	4.02E-05	87.91818147
16	400	3	5	502.4	1.63E-04	75.74727066

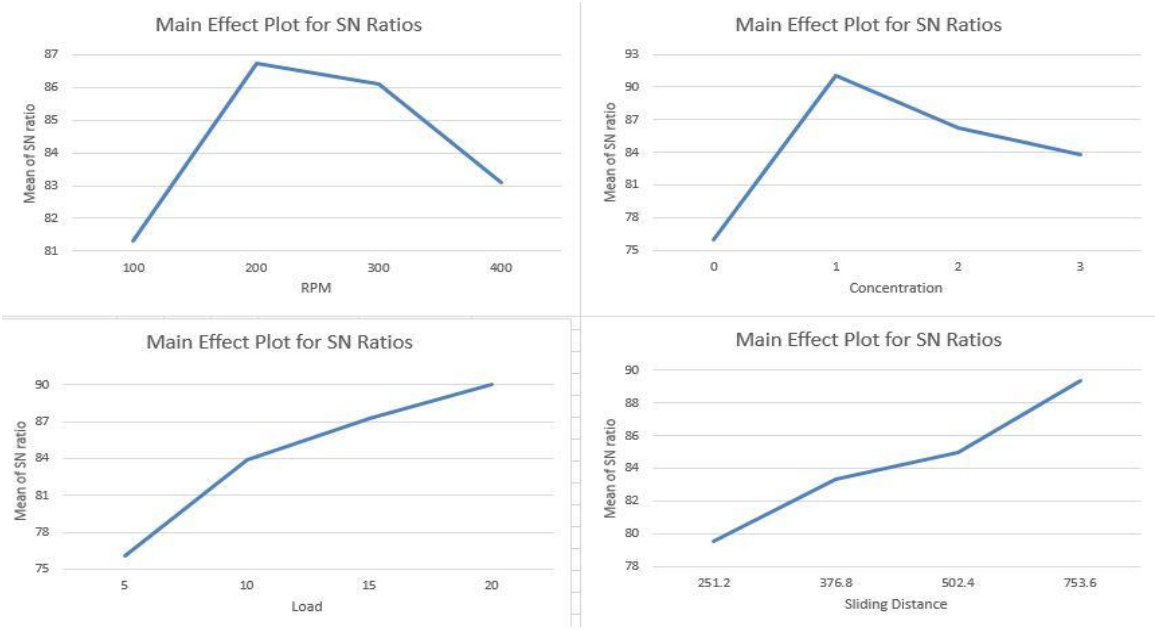

Figure 30 Specific wear rate for pristine MWCNT composite.

Table 10 L_{16} Orthogonal Array for functionalized MWCNT composite, specific wear rate

S. No.	RPM	Concentration	Load	Sliding Distance	Specific wear rate	SNRA1
1	100	0	5	251.2	1.21E-03	58.30880822
2	100	1	10	376.8	7.09E-06	102.9818673
3	100	2	15	502.4	1.30E-05	97.70524312
4	100	3	20	753.6	1.13E-05	98.9480806
5	200	0	10	502.4	1.11E-04	79.08716954
6	200	1	5	753.6	7.09E-06	102.9818673
7	200	2	20	251.2	1.42E-05	96.9494719
8	200	3	15	376.8	3.01E-05	90.42870595
9	300	0	15	753.6	4.71E-05	86.54782268
10	300	1	20	502.4	5.32E-06	105.480642
11	300	2	5	376.8	5.21E-05	85.66404329
12	300	3	10	251.2	5.70E-05	84.87772795
13	400	0	20	376.8	9.72E-05	80.24700848
14	400	1	15	251.2	2.13E-05	93.43944222
15	400	2	10	753.6	1.66E-05	95.61053611
16	400	3	5	502.4	7.49E-05	82.51574171

Fig. 31 represents effect of various control factors on the specific wear rate for functionalized MWCNT composite, for this specific wear rate comes out to be minimum

for 1F composite at 200rpm, 1wt% concentration of functionalized MWCNT composite, at a loading of 20N and sliding distance at 753.6 m.

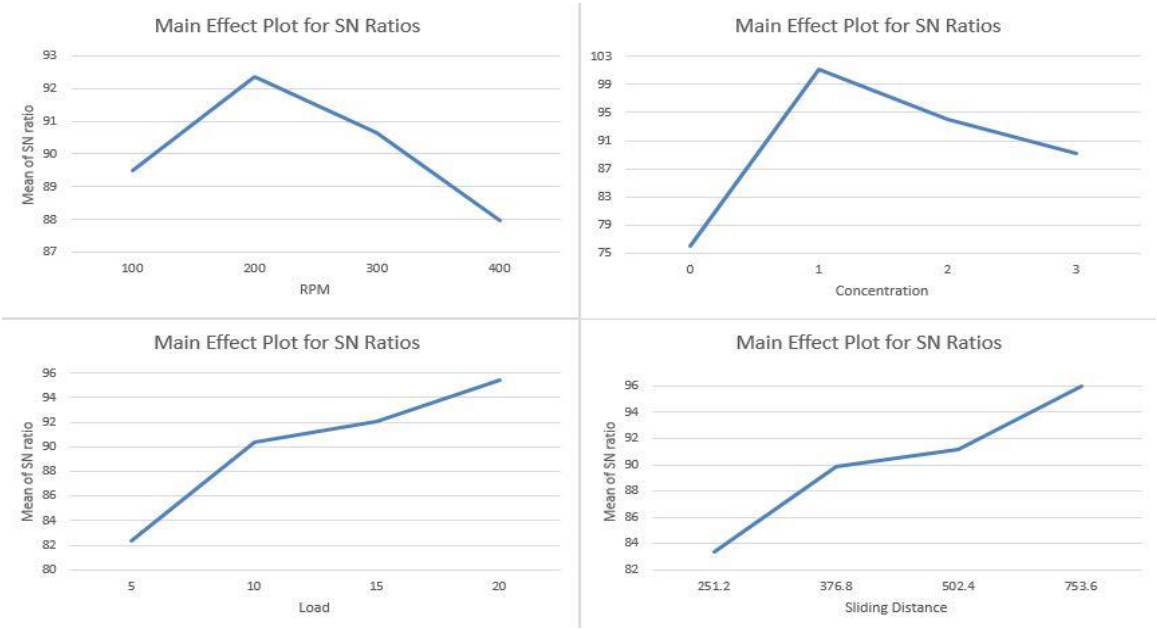

Figure 31 Specific wear rate for functionalized MWCNT composite.

As explained above at higher loading percentage the bonding strength decreases because of that on increasing the concentration wear rate increases. But, the specific wear rate for functionalized MWCNT composite is less than the pristine MWCNT composite as because of functionalization interfacial bonding increases and thus wear resistance.

4.3.2 ANOVA and the Effects of Factors

Analysis of Variance has been done from the sliding wear data of pristine MWCNT composite and functionalized MWCNT composite. In order to find out statistical significance of various factors viz. RPM, concentration, load, Sliding distance Table 11 and Table 12 shows the ANOVA result for specific wear rate for pristine MWCNT composite and functionalized MWCNT composite respectively. The last column of the table indicates percentage contribution of the control factors and their interactions on the performance output i.e. specific wear rate. Analysis is done for a level of confidence of significance of 5%.

From Table 11, it can be observed for pristine MWCNT composite that concentration (p = 0.01), load (p = 0.012) and sliding distance (p=0.035) have considerable influence on specific wear rate. The interaction of RPM (p=0.116) have relatively less significant contribution.

Table 11 ANOVA table for specific wear rate

(For pristine MWCNT reinforced epoxy composites)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
RPM	3	78.24	26.079	4.76	0.116
Concentration	3	473.95	157.984	28.83	0.01
Load	3	439.42	146.473	26.73	0.012
Sliding Distance	3	198.43	66.143	12.07	0.035
Error	3	16.44	5.48		
Total	15	1206.48			

** DF: degree of freedom, ## Seq SS: sequential sum of squares, $ Adj. SS: extra sum of squares

$$ Seq MS: sequential mean squares, *** F: F-test, ### P: percent contribution

Similarly, from Table 12 it can be observed for functionalized MWCNT composite that concentration (p = 0.013) have major influence on specific wear rate. The interaction of RPM (p = 0.596), load (p = 0.079) and sliding distance (p = 0.092) have relatively less significant contribution.

Table 12 ANOVA table for specific wear rate

(For functionalized MWCNT reinforced epoxy composites)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
RPM	3	41.58	13.86	0.74	0.596
Concentration	3	1348.15	449.38	23.91	0.013
Load	3	367.85	122.62	6.52	0.079
Sliding Distance	3	325.28	108.43	5.77	0.092
Error	3	56.39	18.8		
Total	15	2139.25			

** DF: degree of freedom, ## Seq SS: sequential sum of squares, $ Adj. SS: extra sum of squares

$$ Seq MS: sequential mean squares, *** F: F-test, ### P: percent contribution

4.3.3 Wear surface morphology

Sliding wear test is done at different concentration and after that optical image at various concentrations are taken and the wear surface morphology is studied. Table 13 and Table 14 shows the specific wear rate and at different concentration for the pristine composite and functionalized composite. The graph between specific wear rate vs. concentration are plotted for pristine MWCNT composite as given in Figure 32. For functionalized MWCNT composite it is given in Figure 33. Trend can be seen as first the specific wear rate decreases on increasing concentration to about 0.5 wt.% and then it increases constantly with concentration. This trend is similar for both pristine and functionalized MWCNT composite, this is so because with the increase of concentration the viscosity of epoxy matrix increases which leads to incomplete matrix infusion. Increase in void content of CNT composite with concentration is also because of the same reason. But, when specific wear rate and for functionalized MWCNT composite is compared with the functionalized MWCNT. The functionalized MWCNT composite showed better wear resistance as after functionalization the interfacial bonding with epoxy matrix increases which in turn has increased the wear resistance.

Table 13 Specific wear rate at different concentration for pristine MWCNT composite

S. No.	Concentration	W1	W2	W1-W2 (g)	Force	RPM	sliding distance	Density	Specific wear rate
1	0	1.96	1.32	0.64	10	200	314.15	1.146803	1.78E-04
2	0.5	2.13	1.91	0.22	10	200	314.15	1.123574	6.23E-05
3	1	2.16	1.92	0.24	10	200	314.15	1.122286468	6.81E-05
4	1.5	2.09	1.84	0.25	10	200	314.15	1.120763429	7.10E-05
5	2	2.25	1.96	0.29	10	200	314.15	1.116829162	8.27E-05
6	3	2.11	1.76	0.35	10	200	314.15	1.114788794	9.99E-05

Table 14 Specific wear rate at different concentration for functionalised MWCNT composite

S. No.	Concentration	W1	W2	W1-W2 (g)	Force	RPM	sliding distance	Density	Specific wear rate
1	0	1.96	1.32	0.64	10	200	314.15	1.146803	1.78E-04
2	0.5	1.22	1.09	0.13	10	200	314.15	1.126388	3.67E-05
3	1	2.52	2.36	0.16	10	200	314.15	1.125855383	4.52E-05
4	1.5	2.19	1.97	0.22	10	200	314.15	1.126514269	6.22E-05
5	2	2.66	2.41	0.25	10	200	314.15	1.122657032	7.09E-05
6	3	2.49	2.15	0.34	10	200	314.15	1.122281307	9.64E-05

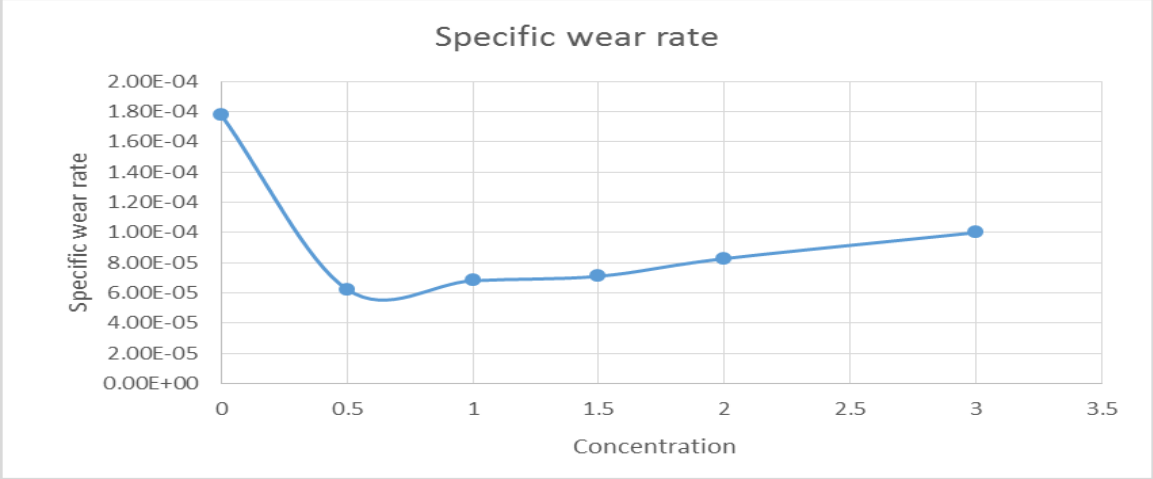

Figure 32 Specific wear rate for pristine MWCNT composite

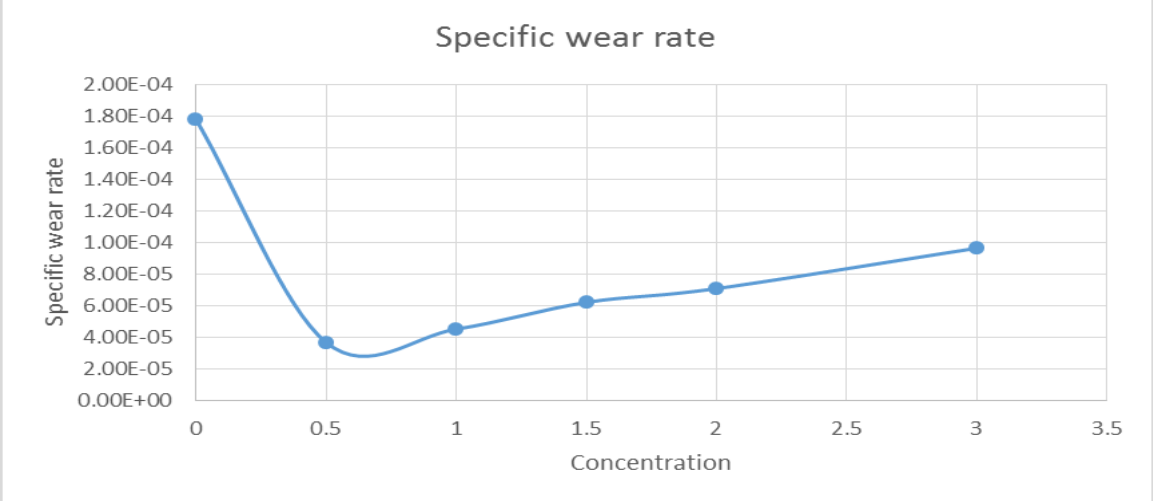

Figure 33 Specific wear rate for functionalized MWCNT composite

As in earlier sections it is learned that specific wear rate is minimum at 0.5wt% concentration. Therefore, optical images for pure epoxy, 0.5wt% pristine MWCNT composite and 0.5wt% functionalized MWCNT composite are taken at 10x and 20x magnification and then are analysed. Figure. 34, 35, 36 shows the wear surface morphology after sliding wear test for neat epoxy, pristine MWCNT composite at 0.5wt% concentration, functionalized MWCNT composite at 0.5wt% concentration respectively.

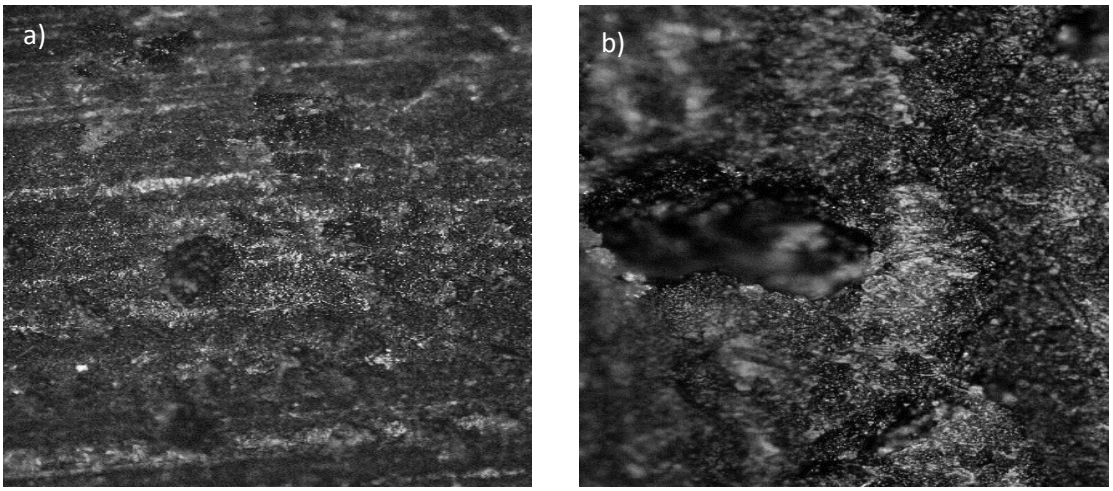

Figure 34 Wear surface morphology of neat epoxy specimen at a) 10x and b) 20x magnification.

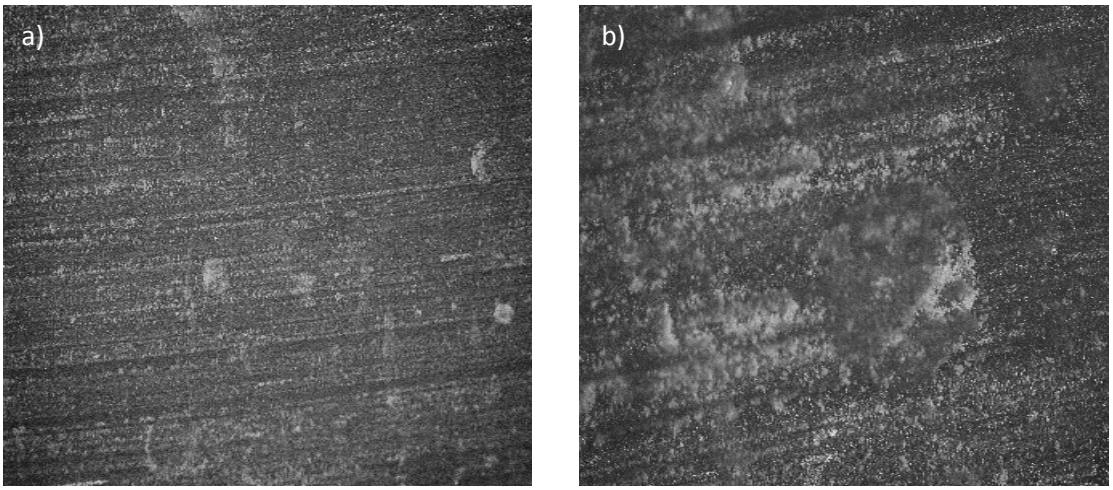

Figure 35 Wear surface morphology of P-MWCNT composite at a) 10x & b) 20x magnification.

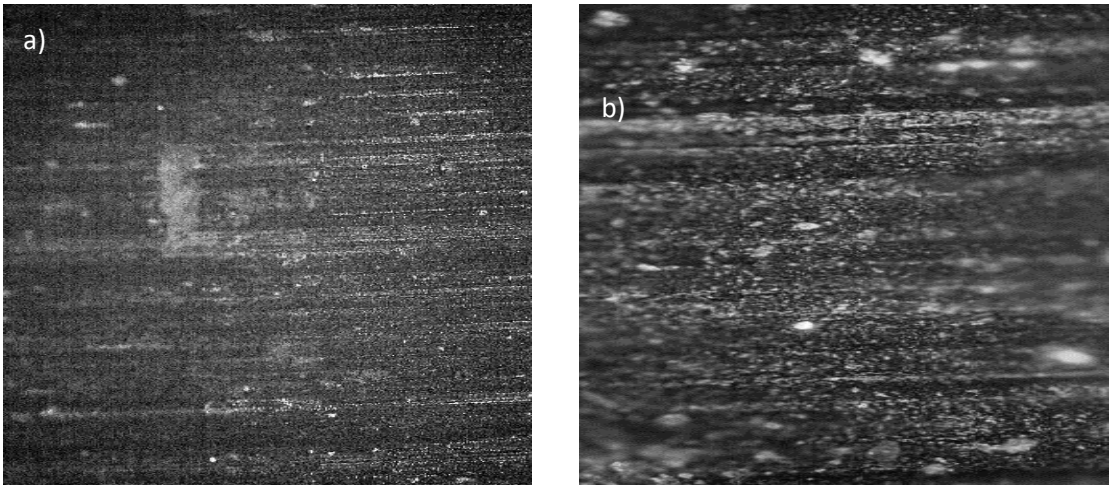

Figure 36 wear surface morphology of F-MWCNT composite at a) 10x & b) 20x magnification.

In pure epoxy and pristine MWCNT composite rough surface and deeper grooves can be clearly seen as compared to the functionalized MWCNT composite. For, functionalized MWCNT composite a smooth profile is seen. From this worn surface morphology it is clear that at the surface exposed CNT acts as a lubricant by providing a thin layer. Thus increasing the wear resistance. But, for the pristine MWCNT composite as the interfacial bonding is lower as compared to functionalized MWCNT composite thus CNT easily comes out of the surface thereby creating a burrs on the surface of composite. Epoxy has deeper grooves than the pristine MWCNT composite. Therefore, it can be concluded that functionalized MWCNT composite has the best wear resistance as compared to pristine MWCNT composite and neat epoxy.

Chapter 5

Summary and Conclusion

- Conclusion
- Application
- Scope for future work

Chapter 5 - Summary and Conclusions

In design of an application based product its lifecycle is foremost criterion and information about tribological properties provides essential evidence towards selection of a product. To this end, MWCNT's were first functionalised by the carboxyl group by the help of chemical functionalization and then are characterized by FTIR, XRD, TGA, DSC, and SEM. Nano composites is fabricated by sonication and stirring and wear test is conducted at various concentration for pristine Nano composites and functionalised Nano composites. Wear surface morphology is studied by optical microscope. Wear tests were conducted on the neat epoxy, pristine MWCNT, functionalized MWCNT. Pristine MWCNTs is functionalised by carboxyl group. Composite materials are prepared with different concentration by pristine multi walled carbon nanotube and functionalised multi walled carbon nanotube. The following conclusions are derived from this study:-

1. Parameters for functionalization of MWCNT were found to be at an acid ratio of 3:1 in a 100 ml mixture acid, temperature at 120^{o}C, time during which solution is heated is 30 minute, and 1g of MWCNT is used for the functionalization.
2. FTIR confirmed the grafting of carboxylic group on to the walls of multi walled carbon nanotubes.
3. Condition of chemical functionalization was not harsh as the structure of graphite is retained as confirmed by XRD.
4. Optimum parameters for reducing the specific wear rate are obtained at 1 wt% concentration, 200 rpm, 20 N load, and at a sliding distance of 753.6 m by Taguchi design of experiments for both pristine MWCNT composite and functionalized MWCNT composite.

5. Specific wear rate for functionalized MWCNT composite is lower than that of pristine MWCNT composite thus functionalized MWCNT composite has higher wear resistance.
6. Significant parameters for pristine MWCNT composite are concentration, load and sliding distance, for functionalized MWCNT composite concentration is found to be the most significant parameter. As, analysed by the ANOVA.
7. Specific wear rate is lower at 0.5 wt. % concentration keeping rpm, load, and sliding distance at 200 rev/min, 10 N, and 314.15 m respectively.

5.1 Recommendation for Potential Applications

Carbon nanotube reinforced epoxy matrix composite fabricated in this research work found to have adequate potential for wide variety of applications in accordance to tribology point of view. Some of them may include manufacturing of light weight sports equipment, wind turbine blades, nozzle, aircraft brake, disk and linings, ultra-light weight aerospace structures, gears, bearings etc.

5.2 Scope for future work

There is a wide scope for future investigators to explore many other aspects of current research work. Some recommendations for future research include: -

1. Study of sliding wear behaviour of functionalized composite by doing functionalization by amide group.
2. Study of response of composite in study to other wear modes of erosion and abrasion.
3. Nano composite may be combined with hybrid composite to arrive at variety of new results.
4. For use in industrial mass production, after analysing their cost effects according to their application.

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